

**STUDIES ON FLOTATION KINETICS OF COPPER
MINERALS FROM MALANJKHAND ORE
USING OPEN TOP LEEDS CELL**

by

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DEPARTMENT OF METALLURGICAL ENGINEERING

INDIAN INSTITUTE OF TECHNOLOGY KANPUR

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INDIAN INSTITUTE OF TECHNOLOGY KANPUR

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Certified that this work "Studies on Flotation Kinetics of Copper Minerals from Malanjkhand Ore Using Open Top Leeds Cell" has been carried out under my supervision and that it has not been submitted elsewhere for a degree.



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TO

BRINDA

RAJESH

and

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ABSTRACT

Semi-batch experiments were carried out on flotation of copper minerals from an ore sample received from Malanjkhand Copper Deposit. Leeds open-top cell, which provides excellent control over impeller speed, air flow rate, pulp level and froth scraping depth, was used for these studies. The cell gave highly reproducible results with less scatter.

Detailed studies were made on the effect of various process and control variables such as grinding time, particle size, pH, collector and frother quantities, impeller speed, and aeration rate on flotation behaviour. The flotation recovery was optimal in the size range -100+200 mesh, decreasing considerably in both coarser and finer sizes. In the -200 mesh size fraction, the recovery was only about 70 percent explaining why the recovery sharply decreased with increasing grinding time. The copper recovery improved with increase in pH. Similarly increase in collector and frother quantities also increased the recovery. In the case of rpm, there was an increase in copper recovery initially, which got stabilised afterwards. Similarly as aeration was increased the copper recovery showed an improvement.

The experimental results were analysed using a kinetic model with distributed rate constant. Estimation of the flotation rate distribution was carried out using a method which involved numerical inversion of Laplace transform using Lobatto quadrature formula. The results are presented and discussed in the light of how the cumulative flotation rate distribution is affected under various process and equipment conditions.

Experiments conducted simulating the rougher-scavenger-cleaner stages showed how the flotation rate distributions varied in all these stages.

A detailed literature survey on flotation kinetics is also presented.

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CHAPTER 1

INTRODUCTION

Froth flotation is one of the major achievements of modern times in the field of extractive metallurgy. Since its inception in the early part of twentieth century, it has been responsible for overwhelmingly increased production of non-ferrous metals. It has made possible the mining of finely disseminated low grade complex ores which were once considered worthless for economic exploitation.

Froth flotation operations involve a complex stochastic interaction between multiphase processes such as particle suspension and dispersion, bubble formation and distribution, bubble particle collision, adhesion and levitation; particulate transport into froth and return to pulp and product removal. The phenomena are not yet completely understood and the history of flotation since its beginning is marked by the use of empirical 'rules of thumb' for process control, scale-up and design. Such a procedure always carries the risk that failure probability grows rapidly with the scale of operation (Harris, 1973).

The quantitative understanding of such an important process is therefore essential, which would lead to improvements both in metallurgical results and flotation cell design. It also should lead to reliable methods for scaling up of laboratory and pilot plant results to full size operations without the need

for expensive and time consuming mill tests on commercial machines. Additionally this will also assist the automation of flotation cells and circuits as well as control by computers (Arbiter and Harris, 1962).

Considerable research work on various aspects of flotation has of course been going on and the flotation process has indeed graduated from the state of being an art to a full fledged engineering science. Although attempts have been made to study the mechanistic aspects and fundamental physical chemistry of flotation process, kinetic models have proved more useful for process simulation and optimisation. Attempts have been made with limited success to set up theoretical models for flotation and derive corresponding rate expressions from first principles.

Most of the kinetic models are derived by applying the concepts and techniques of chemical reaction engineering (Arbiter and Harris, 1962) for a phenomenological analysis of flotation systems. While the use of analogue models may be questioned on theoretical grounds (Mika and Fuerstenau, 1968), the kinetic description has been successfully used in simulation and optimisation of flotation plants (King, 1976). On account of the fact that the feed material comprises of billions and billions of particles varying in size, mineral content, hydrophobicity etc., it is but rational to associate a distributed rather than a single valued rate constant with the feed material. In literature, one can find many mathematical models incorporating

this feature (Kelsall, 1961; Pogorely, 1961; Imaizumi and Inoue, 1963; Woodburn and Loveday, 1965; Inoue and Imaizumi, 1968; Mehrotra, 1973; Kapur and Mehrotra, 1973). However, a rigorous study to analyse the dependence of the apparent flotation rate constant distribution on different process and equipment variables has not been made yet. Present thesis is an attempt in the direction of filling this gap.

Before doing the experimental work an extensive review of literature on flotation kinetics was carried out. This is summarised in Chapter 2 of this thesis. Semi-batch experiments were carried out on an ore sample from Malanjkhand Copper Deposit, under various experimental conditions, using Leeds Open Top flotation cell. The variables studied included particle size, pH, collector and frother quantities, impeller speed and aeration rate. The details of raw materials used, experimental set-up and experimental procedure are presented in Chapter 3. The results are presented and discussed (Chapter 4) in the light of how the cumulative rate constant distribution is affected under various process and equipment conditions.

CHAPTER 2

FLOTATION KINETICS - A REVIEW

Extensive work is reported in literature on the study of flotation process. This technique has been studied from many angles. Adsorption, contact angle, infrared and other such studies have thrown light on the physico-chemical factors affecting flotation while modelling and kinetic studies have led to a better understanding of the process and reasonable predictions (Harris, 1973). In addition, considerable work has been done on flotation network synthesis and optimisation, scale-up and design of big industrial cells, automatic control of flotation circuits etc. It is beyond the scope of this present study to cover all these areas. So a brief review is presented below on modelling and kinetic aspects of flotation, which pertains to the present study.

The mathematical description of flotation as a rate process was first given by Garcia-Zuniga (1935), who on the basis of experimental data in a semi-batch process, reported that the material remaining in the pulp decays exponentially with time. Since then a number of kinetic equations and models have been proposed. In this review, these models have been discussed in more or less the same chronological order, which is reflected in the increasing degree of their sophistication as we proceed from one model to the other.

2.1 KINETIC MODELS BASED ON CHEMICAL ANALOGY

Taking the analogy from the chemical reaction kinetics Beloglazov (1939) proposed that the rate of decrease of material in pulp with time is proportional to the amount of material in pulp at that instant, i.e.

$$\frac{dC(t)}{dt} = -K_a C(t) \quad (2.1)$$

where K_a is the apparent flotation rate constant and $C(t)$ is the concentration of solid particulate species in pulp. The first order kinetic form probably led Grunder and Kadur (1940), and later Plaksin et al (1956) to adopt the law of mass action in chemical kinetics for their equations. A more general rate expression was given by Arbiter and Harris (1962) as

$$\frac{dC(t)}{dt} = -K' \prod_{i=1}^N C_i^{n_i}(t) \quad (2.2)$$

where C_i is the concentration in pulp of i^{th} species, n_i is the order of reaction with respect to i^{th} species, and N is the total number of species in the system. Assuming only two constituents in the pulp, namely solid particles and air bubbles, and air supply to be constant, Eq. 2.2 reduces to

$$\frac{dC(t)}{dt} = -K_a C^{\bar{n}} \quad (2.3)$$

where K_a and \bar{n} are the overall rate constant and order of reaction respectively. Schuhmann (1942) pointed out that for $\bar{n} = 1$, the rate constant K_a has the same dimensional significance as

that for the first order chemical rate equation. However, Mika (1969) pointed out that the analogy between flotation and chemical reaction rate equations arises from the use of the terms, such as rate constant and order of reaction and otherwise these parameters as such have no physical significance except when $\bar{n} = 1$.

There has been some controversy in the literature regarding the value of \bar{n} in Eq. 2.3. Unity for \bar{n} was recommended by Garcia-Zuniga (1935), Beloglazov (1939), Grunder and Kadur (1940), Schuhmann (1942), Volkova (1946), Sutherland (1948), Morris (1951), Brown and Smith (1953), Jowett and Safvi (1960), Modi and Fuerstenau (1960), Imaizumi and Inoue (1961), Chi and Young (1962), and Kapur and Mehrotra (1973), whereas Arbiter (1951), Hukki (1953), Mitrofanov (1953, 1954), Bennett et al (1958) and Suwanasing and Salman (1970) proposed a value of two. Various other values have been assigned to \bar{n} by Bogdanov et al (1954), de Bryun and Modi (1956), Horst (1956, 1958) and Volin and Swami (1964). Plaksin et al (1956) asserted that it would be incorrect to assume a constant value for \bar{n} during the entire period of flotation, because conditions and forms of the mineralized bubbles undergo change during the process. Kakovskii (1954) and Krokhnin (1956) divided the whole process into several stages and described each stage by a separate equation.

Examination of non-first order systems led Pogorely (1962) and Tomlinson and Fleming (1965), to introduce the concept of free and inhibited flotation. Free flotation results when the bubbles are only sparsely loaded with mineral particles

and this can be described by first order rate equation since it is likely that conditions and forms of mineralized bubbles remain constant. In inhibited flotation bubbles are saturated with mineral particles to the extent determined by the surface capacity to accommodate particles, or its lifting power.

Pogorely (1962) gave generalised equation for both the systems, which reduces to first and zeroth orders in low and high concentrations of solids in pulp respectively.

Although treatment of Eq. 2.3 with \bar{n} as greater than unity has been reported by many workers, the first order treatment ($\bar{n} = 1$) received the widest approval. The integrated form of Eq. 2.3 is given as

$$C(t) = C(0) e^{-K_a t} \quad (2.4)$$

where $C(0)$ is the initial concentration of material in the pulp. According to this, a plot of $\ln(C(t)/C(0))$ versus time t should be a straight line, which is seldom the case. This could be due to the implicit assumption made in deriving Eq. 2.4 that the solids in the feed are homogeneous, i.e., all of them possess similar flotation properties. Morris (1952) modified the Eq. 2.3, incorporating an additional parameter C_∞ , as

$$\frac{dC(t)}{dt} = -K_a (C(t) - C_\infty) \quad (2.5)$$

and Bushell (1962) justified C_∞ with the argument that the feed contained in addition to floatable material, a certain amount of material that would remain in pulp without floating even

after infinite flotation time. This fraction of material represented by C_{∞} , has $K_a = 0$. Similar observations were also made by Cooper (1966). On the other hand, Jowett and Safvi (1960) pointed out that C_{∞} was merely an empirical parameter, whose actual behaviour did not correspond to the true terminal concentration and, moreover, instead of being strictly a mineralogical variable, it apparently represented the hydrodynamic nature of the test cell as well as the flotation chemistry.

Extending the argument further the flotation feed could be assumed to contain material with varying K_a values. This becomes the basis of distributed rate constant models, which will be discussed later.

Krokhin (1975, 1980), developed a kinetic model and simulated the batch flotation of mixtures of quartz, galena, schellite, barite etc. He also supported the earlier view of Plaksin et al (1956) that the flotation rate constant decreased during the course of a flotation test by a factor of 2-5, and the lack of linear relation was due to size difference of floatable material. Xu (1985), using a kinetic model, could arrive at the required tank arrangement, column height etc.

2.2 PROBABILISTIC AND STOCHASTIC KINETIC MODELS

Schuhmann (1942) introduced probabilistic concept in describing the flotation process. The overall probability of flotation of a particle, P_o , may be expressed in terms of the probabilities of collision (P_c), adhesion (P_a) and the particle being retained by the bubble (P_r). If all these probabilities

are assumed to be stochastically independent, then the overall probability, P_o is given as

$$P_o = P_c \cdot P_a \cdot P_r \quad (2.6)$$

The probability P_r was modified by Tomlinson and Fleming (1965), as a product of two other probabilities P_e and P_f , the probability of levitation of the mineralized bubble from the pulp to froth phase and the probability of drainage from the froth respectively. Rewriting Eq. 2.6,

$$P_o = P_c \cdot P_a \cdot P_e \cdot P_f \quad (2.7)$$

The major drawback of this treatment is the questionable assumption of stochastic independence of all sub-probabilities. Similar approach was however, adopted by many workers (Bogdanov, 1954, 1965; Kelsall, 1961; 1974, Davis, 1964). A simple form of probability model was used by Kelsall for a battery of continuously operated cells in series. The mass of material M_T , leaving in tailings after passing through n compartments in series was given as,

$$M_T = M_F(1 - P_o)^n \quad (2.8)$$

where M_F is the mass of material in feed. Kelsall also suggested a graphical procedure to evaluate P_o . Davis applied this approach to model industrial plants floating lead and zinc sulphide minerals. However as pointed out by Woodburn (1970), the main objection to this model was that it failed to take

into account the increase in residence time per cell down a consecutive cell bank, which resulted in gradual increase in probability of flotation.

Bodzinoy (1965) developed a stochastic model for flotation, according to which the flotation could be described as a so-called "Simple Death Process", which reduces to the first order kinetics in the mean. Katz and Shinnar (1969) considered flotation as a random phenomena belonging to the family of Markov processes.

It can be shown that the simple form of probability approach is similar to the simplest form of the kinetic model. Even in delineation of more complex probability models and kinetic models, an area of overlap exists (Lynch et al, 1981).

2.3 MULTIPHASE MODELS

Questioning the oversimplification by analogy to chemical kinetics, of a complex process such as flotation, involving separation of species in more than a single phase, Arbiter and Harris (1962) proposed a kinetic model, in which the cell contents were partitioned between two phases of pulp and froth. The two phases were assumed to be ideally mixed, so the tailings provided a representative sample of the pulp and the concentrate, of the froth. Material transport occurred across the boundary between the two phases in one or both directions. Any order of kinetics could be accommodated (Harris and Rimmer, 1966), though first order kinetics was most favoured.

According to this model, mass flow-rate balance relationships can be written separately for pulp, froth phases and

concentrate removal, respectively as follows:

$$\frac{dM_p(t)}{dt} = -k M_p(t) + h M_f(t) \quad (2.9)$$

$$\frac{dM_f(t)}{dt} = k M_p(t) - (h + 1) M_f(t) \quad (2.10)$$

and
$$\frac{dM_c(t)}{dt} = l M_f(t) \quad (2.11)$$

where M_p , M_f and M_c are masses of floatable solids at time t , in pulp, froth and concentrate respectively. k , h and l are rate constants for material transfer from pulp to froth, froth to pulp and froth to concentrate respectively.

Solutions to the above three equations have been reported for numerous special cases (Arbiter and Harris, 1962; Harris and Rimmer, 1966) including semi-batch; continuous flotation; step changes in input and output; no return from froth, $h = 0$ and no removal of concentrate, $M_c = 0$.

Although this model was based on the questionable assumption that the froth phase also was perfectly mixed, it was, nevertheless, successfully used by Harris et al (1963) to correlate experimental data on continuous flotation of coal at steady state. Bull (1966) and Bogdanov et al (1970) have concurred with this approach. Ball and Fuerstenau (1970) and Ball, Kapur and Fuerstenau (1970) have introduced the concept of distributed rate constant into this model. They tried to determine the rate constants for material transfer from pulp to froth and froth to pulp and to distinguish the flotation models

with distributed and non-distributed rate constants (Ball and Fuerstenau, 1974).

The two-phase model could be extended into a multiphase model by dividing the pulp and froth into several mixed phases, each with two-way interchange with its neighbours, allowing step-wise concentration gradient from phase to phase in the froth (Harris, 1966). This model was opposed by many workers (Niemi, 1966a; Woodburn and Loveday, 1966; Imaizumi and Inoue, 1965) on the ground that for sufficiently fast rate of froth removal, the rate of return of material from froth to pulp was negligible and therefore two-phase models were not necessary. However, this point was criticised by Maksimov and Khainman (1965), who observed that even for high concentration yields and with the use of reagents which ensured a stable froth, the probability of particles remaining in the froth was much smaller than unity and the drainage rate increased with particle size.

The two-phase model got wide acceptance on modelling of flotation circuits, because of its ability to focus on the critical froth phase and because of the ease with which its mathematical expression could be incorporated into circuit equation (Woodburn et al, 1976). This model was not very good with respect to scale-up and it needs modification in order to describe transient effects in detail. A lot of work is being reported on the various sub-processes taking place in the pulp and froth phases. Transport behaviour in the two-phases, froth structure, entrainment and drainage have all received much

attention recently. An exhaustive review of this model was done by Harris (1978).

Sadler (1973) derived a transfer function solution to the two-phase model. Since he considered the simple two-phase model unacceptable for predicting continuous cell dynamics, he developed a "bubble surface area model". Mass transfer in this model was based on the amount of bubble surface available for particle attachment and its rate of introduction into the cell. Watson and Allen (1973, 1974) reported that froth height and the ratio of the masses in the froth and in the pulp, both varied linearly with airflow rate.

Greaves and Allen (1974) also supported the view of Harris et al (1963) that the rate constant h , in Eq. 2.9 and 2.10 was proportional to the feed concentration. They studied the two-phase model under steady state and dynamic conditions. Description of the steady state was generally satisfactory, but the model did not describe the transient state well. Cutting and Davenish (1975) developed a model of froth structure using a steady state type batch cell, which can be used to predict the performance of continuous flotation process, from batch tests and also to predict changes in recovery and other factors, when changes in froth level are made.

Many authors considered different mixing regimes in the froth phase. Bushell (1962) proposed that the rate of particle return from froth to pulp was proportional to the rate of particle transfer in the reverse direction. Cooper (1966) and Niemi (1966b) considered the froth phase as the plug-flow regime,

contrary to the assumption of the two-phase model. They treated the froth phase to be horizontally homogeneous, but vertically distributed. Mika (1967) compared both the plug-flow and mixed flow treatments and showed that under certain conditions, Cooper's model reduced to two-phase model. Moys (1973, 1984) proposed models incorporating froth features such as froth stability, froth removal rate and the residence time ratio of the 'perfectly floatable' particles. Cutting et al (1981) observed that from batch data, the material transfer from pulp to froth could be estimated and the static froth concentration could be determined using their "equilibrium cell". They have discussed in detail (Cutting et al, 1982) the importance of froth structure in the prediction of plant performance using process models.

The pulp phase was also treated as being made up of more than a single phase by Harris et al (1975) and Harris (1976a,b). Residence time measurements performed on laboratory machines showed deviations from ideal mixing. The impeller region was intensely mixed and it was wholly contained within the outer region which, though less intensely mixed, still conformed to ideal mixing. Reversible interchange occurred between the two regions, but flow into and out of the cell occurred only via the outer region. Harris (1976b) discussed another model also, in which feed entered into the system via the impeller region. Feteris et al (1987) combined the probability and two-phase models to derive a relation between the

overall flotation rate coefficient and probability that a particle-bubble aggregate survives the cleaning action of froth, which depends on the depth of the froth phase.

2.4 KINETIC MODELS BASED ON MECHANISTIC APPROACH

Mechanistic models break the complex flotation process into a number of sub-processes taking place in the cell competitively or consecutively. The mechanisms of the sub-processes are treated from the basic physico-chemical principles. Although some of these models are fairly complex to be of much industrial utility at the present moment, nonetheless, they provide an insight into the likely mechanisms of the process.

Mineral particles are transferred from pulp to froth through one or more of the following four mechanisms:

- (1) Attachment of the mineral particles with the air bubbles in the pulp followed by transfer of bubble-particle assembly to the froth.
- (2) Mechanical straying of the mineral particles into the froth column.
- (3) Selective precipitation of the gas dissolved in water on the surface of mineral particles, and
- (4) Attachment of the mineral particles to the bottom of the froth column by agitation of the pulp.

The last two mechanisms are not very important and the major fraction of the material transfer is through bubble-particle aggregates.

There has been a certain amount of controversy on the mechanism of particle-bubble attachment. One school of thought

(Taggart, 1948) is that the bubbles grew on the surface of the particle after getting precipitated from the water. This has been criticised by Bogdanov and Filianovski (1940), and Gaudin (1932), who advocated "collision theory" in which bubbles passing through the pulp collide with particles and coalesce with them if the surface conditions are appropriate. Strong support for the collision theory was provided by the experiments of Malozemoff and Ramsey (1941), Plante and Sutherland (1944), Spedden and Hennan (1948), Whelan and Brown (1956), Kirchberg and Topfer (1965) and Imaizumi and Inoue (1965). Klassen (1945) proposed an intermediate mechanism in which a large bubble coalesced with a microbubble precipitated and attached to mineral surface. Smith et al (1957) and Klassen (1960) have reviewed the conditions under which gas precipitation on the mineral surface becomes important.

The capture of a particle by a bubble can be thought of as occurring in a series of sequential stages, namely adsorption of reagents on the surface of particles, collision between particles and bubbles, attachment, partial detachment of particles from the bubbles due to gravitational and inertial forces, froth instability etc. The kinetic models developed on the basis of mechanistic approach incorporate one or more of these steps.

First such model was put forth by Sutherland (1948), in which the overall rate of flotation was equal to the product of three factors: the rate of collision between particles and bubbles; the probability of adhesion after collision; and the

probability that detachment would not take place subsequently. Assuming water is viscous and incompressible, and that particles and bubbles are both rigid spheres and incompressible, he calculated the first two factors. If the radius of the particle is r_p , a stream tube could be chosen that surrounds the bubbles as it rises and passes within the distance r_p from the equator. All particles within the stream tube ahead of the bubble are then potential candidates for capture. The radius of the stream tube at infinite distance from the bubble was defined as the "collision radius", r_c , and was given as

$$r_c = \sqrt{3r_b r_p} \quad (2.12)$$

where r_b is the radius of the bubble. Photographic observation by Spedden and Hennan (1948) showed that it was possible for larger particles striking the bubble to deform it elastically. This deficiency was removed by Philippoff (1952) and Evans (1954) who independently postulated a bounce theory in which role of inertial forces was emphasised. Further modification to this theory was proposed by Whelan and Brown (1956). On the other hand, empirical correlation reported by Langmuir (1948), Herne (1960), Ranz and Wang (1952) on collision of particles seem to be consistent with the results of Sutherland. Similar approach was adopted by Bogdanov et al (1965) to calculate the probabilities of collision and adhesion. Derjaguin and Dukhin (1961) regarded the approach of a particle to a bubble as occurring in three zones: the zone 1 being outside the range of molecular and diffusional forces, contained particles acted upon purely by hydrodynamic forces; the zone 3 comprises the area very close

to the surface of the bubble in which forces of molecular origin predominated; the zone 2 was between the zones 1 and 3 in which diffusional effect could be important. They showed that the particles in this region experienced a 'diffusiophoretic' force. Lyman (1974), while acknowledging the existence of this force, showed that with simple Gouy-Chapman theory of double layer, the diffusiophoretic contribution vanished. Flint and Nowarth (1971) evaluated the collision efficiency of a particle with a bubble from the consideration of a particle trajectory as a function of particle size and hydrodynamic conditions prevailing in the cell. Although this study provided a qualitative understanding of the collision phenomena, its extension in quantitative sense, to a system of swarm of bubbles was not satisfactory. Reay and Ratcliff (1973) also calculated the collision efficiency of a very fine particles wherein Brownian diffusion would be the dominant capture mechanism. However these two works suffer because of the oversimplification of the hydrodynamics. Collins (1975) modified the hydrodynamic factors and calculated the collision efficiency. The hydrodynamics of the bubble was studied by many other workers also (Harper, 1972; Levich, 1962).

A rate equation based on analogy between adsorption and mineralization of air bubbles was derived by Matveenko (1957). A more detailed and structurally complex phenomenological model was proposed by Mika and Fuerstenau (1968), wherein each elementary rate process was formulated in terms of linear first

order differential equation for individual discrete size particles and bubble species. Although this model clearly identifies and elucidates the basic steps in interphase transfer of particles, its practical utility remains to be demonstrated. Later Heynman (1975) continued the same argument and considered flotation as a multistage process, taking place in the pulp and froth. However this model had limitations in that it led to complicated equations difficult to solve without making simplifications. Woodburn et al (1971) have proposed a kinetic model for a continuous flotation cell, in which the rate constant was derived as a function of particle size, mineralogy, and the tailings flow rate. Zeidenberg et al (1965) attempted an analysis of the flotation kinetics by use of a multivariate distribution of the particulate entities in particle size, degree of liberation in individual size fractions and floatability for a given size and mineral composition. Kapur and Mehrotra (1973) proposed a kinetic model incorporating the bubble-particle attachment and detachment sub-processes. They treated the flotation feed as being distributed with respect to the bubble-particle attachment rate constant, K and derived an explicit relationship between K and the overall flotation rate constant, K_a . These authors were the first to stipulate conditions under which first order kinetics would indeed be valid. Langler and Hoffmann (1974) developed a mathematical model for flotation similar to the kinetic model of gas adsorption on solids. The rate of flotation depended on the gas-liquid interface and the area covered on the solid. Flint (1974) developed

a model which included material transfer steps in the sub-processes. Rubinshtein and Filippov (1976) derived the probability of a particle collision with a bubble on the basis of binary collision theory by considering the inertial settling and the effect of consumption of collector, the normal component of K.E. relative to the movement of particles and bubbles and a steric factor characterising the relative position of particle and bubble during collision. Aniruns and Kitchener (1976) made measurements of the rate of capture of single particles of strongly hydrophobic, surface-methylated quartz and compared the results with the theoretically calculated values. Rulev et al (1977) derived equations in mono- and poly-dispersed systems based on capture efficiency of a single bubble with regard to the effect of the adjacent ones. Samygin et al (1977) applied a multi-stage model to analyse the experimental data on pyrite flotation. They explained the dependence of floatability on the bubble diameter as being due to the hydrodynamic capture cross-section of particles. Jameson et al (1977) reviewed the various physical factors affecting the flotation process. Meyer and Klimpel (1983) coupled the mechanical removal of froth by paddles with the more rapid physical and chemical interactions taking place in the bath. Szatkowski and Freyberger (1985a,b) constructed a mathematical model based on three important phenomena: collection efficiency, rate of bubble coalescence in flotation pulp and the buoyancy or lack of it, of the mineral-laden bubbles. They argued that the flotation kinetic equations

should not be written in terms of time but on quantity and properties of the air bubbles that are introduced into the pulp. They derived an equation

$$M = M_0 \exp[-(FAF)V_a] \quad (2.12a)$$

where M_0 was the amount of floatable material in pulp before the introduction of air, M was the amount after V_a litres of air was passed, and FAF , flotation air factor, a constant similar to the ordinary flotation rate constant. Laplante et al (1983a,b, 1984) determined the bubble size distribution and flotation rates in a specially designed batch flotation cell, as a function of air flow rates and frother addition. They developed a model considering the effect of bubble size on the total bubble surface area and bubble-particle collision efficiency. Woodburn and Wallin (1984) proposed a model in which the flotation rate constant was decoupled into kinetic effects on the basis of aeration rates, and equilibrium effects that represent the inherent distribution of a solid species between an air interface and the continuous pulp phase.

2.5 KINETIC MODELS WITH DISTRIBUTED APPARENT RATE CONSTANT

It was mentioned in Section 2.1 that just as the flotation feed could have floatable material with $K_a = 0$, then it could also have floatable material with varying K_a values. Since the feed comprises particles distributed in size, mineral content, surface properties, hydrophobicity etc., it is only rational to associate a distributed, rather than a single rate

constant with the feed. This forms the basis for kinetic models under this heading.

The simplest model based on this argument, put forth by Kelsall (1961), visualised two discrete floatable species in the feed, one fast floating and the other slow floating. The mass of floatable material $M_p(t)$, remaining in cell at time t is given by

$$M_p(t) = M_p(0) \left[\phi \exp(-K_{as}t) + (1 - \phi) \exp(-K_{af}t) \right] \quad (2.13)$$

where ϕ is the proportion of slow floating species in the feed and K_{as} and K_{af} are the rate constants of slow and fast floating species respectively. The parameters ϕ , K_{as} and K_{af} can be graphically estimated.

Extending the argument, the feed can be thought to consist of a number of floatable species with different K_a values, such that $M_p(K_{ai})$ is the amount of i^{th} floatable species with rate constant K_{ai} and if $M_p(K_{ai})$ is normalised

$$\sum_{i=1}^n M_p(K_{ai}) = 1 \quad (2.14)$$

where n is the total number of species. Eq. 2.13 now transforms to

$$M_p(t) = \sum_{i=1}^n M_p(K_{ai}) \exp(-K_{ai}t) \quad (2.15)$$

Pogorely (1961) and Imaizumi and Inoue (1965) were among the first authors to formally introduce this concept of variable and distributed rate constant. A method for evaluating K_{ai} and

corresponding $M_p(K_{ai})$ was proposed by Imaizumi and Inoue (1965) and Huber-Panu (1965), which involved taking the asymptotes of the plot of $\ln M_p(t)$ versus t . Unfortunately the number of species n is not known 'a priori' and must be assumed arbitrarily. Complete identification of the discrete distribution from noisy experimental data is a very difficult task. Black and Faulkner (1972) arbitrarily assigned values to n and K_{ai} and evaluated $M_p(K_{ai})$ in Eq. 2.15 by linear regression techniques. This is essentially a curve fitting exercise and provides no insight into the structure of distribution. A large number of different equations can fit the data quite accurately and therefore evaluation of $M_p(K_{ai})$ in this manner is rather meaningless. Many authors (Tille and Panou, 1968; Woodburn et al, 1970; Huber-Panu, 1970) discussed the analysis of experimental data using discrete distributed rate constant models.

The feed can also be thought to be consisting of floatable species continuously distributed in apparent rate constant, such that $M_p(K_a)dK_a$ is an absolute frequency function, explicitly representing the mass of particles with overall rate constant in the range K_a to $K_a + dK_a$. Since the rate of removal of solids from the pulp to froth phase is a time-invariant first order process, one can write

$$\frac{dM_p(K_a, t)}{dt} = -K_a M_p(K_a, t), \quad 0 \leq K_a \leq \infty \quad (2.16)$$

and solution to the above equation is

$$M_p(K_a, t) = M_p(K_a, 0) \exp[-K_a t] \quad (2.17)$$

where $M_p(K_a, 0) = M_p(K_a)$. The total material remaining in cell at time t is found by integrating the Eq. 2.17 with respect to K_a over its full range. That is

$$M_p(t) = \int_0^{\infty} M_p(K_a) \exp(-K_a t) dK_a \quad (2.18)$$

If normalised, $M_p(K_a)$ becomes a true density function and its value ranges from 0 to 1. In order to solve Eq. 2.18, one must estimate the density function $M_p(K_a)$ from noise corrupted discrete experimental data $M_p(t)$. A commonly used method for this purpose consisted of assuming a standard functional form for $M_p(K_a)$ in Eq. 2.18 and determining the parameters of the distribution from the experimental data.

As the simplest case, a rectangular distribution for $M_p(K_a)$ was assumed by Huber-Panu (1964), and if K_a ranges from 0 to a finite maximum value K_{am} , solution to Eq. 2.18 becomes,

$$M_p(t) = \frac{1}{K_{am} t} \left[1 - \exp(-K_{am} t) \right] \quad (2.19)$$

Woodburn and Loveday (1966) and Inoue and Imaizumi (1968) suggested a gamma distribution for $M_p(K_a)$ and evaluated the parameters by graphical methods. Therefore if

$$M_p(K_a) = \frac{\lambda^\alpha K_a^{\alpha-1} e^{-\lambda K_a}}{\Gamma(\alpha)} \quad (2.20)$$

$$\text{then } M_p(t) = \left[\lambda / (\lambda + t) \right]^\alpha \quad (2.21)$$

Loveday (1966) observed that a better fit to experimental data was obtained with incomplete gamma distribution. Ball, Kapur

and Fuerstenau (1970) also used a gamma distribution for $M_p(K_a)$ and solved Eq. 2.18 using moments. Harris and Chakravarty (1970) summarised a large number of distributions for $M_p(K_a)$ and recommended a bimodal gamma distribution. They also pointed out that in Eq. 2.18, $M_p(t)$ is nothing but the Laplace transform of $M_p(K_a)$. Extending this observation further Kapur and Mehrotra (1974) recognized that the problem of finding $M_p(K_a)$ is equivalent to finding the inverse of Laplace transform in Eq. 2.18. Hence in the case of experimental data of $M_p(t)$, the inverse must be computed by numerical means. Unfortunately numerical inversion of Laplace transform is not an easy task, and success is not always assured, especially in the presence of experimental errors. This is because Laplace inverse is a very unstable operator, which implies that arbitrarily small changes in $M_p(t)$ may cause very large changes in $M_p(K_a)$. Although in the literature, a number of algorithms are available for numerical inversion of the exact data, they invariably become ineffective in the case of noise corrupted data. A simple method for the effective numerical inversion of Laplace transform in presence of experimental errors was proposed by Mehrotra (1973) and Kapur and Mehrotra (1974). In this method, the integral in Eq. 2.18 was approximated by the Lobatto quadrature formula and the cumulative rate distribution was evaluated by optimising a least square objective function. Zeidenberg's (1965) model, based on multivariate treatment had a drawback that neither the conditional distribution in mineralogy for a

grain size, nor the floatability as a function of particle size and mineralogy, was known. However, Woodburn et al (1971) showed that even with a limited extension of first order treatment on the lines suggested by Zeidenberg et al offers significant advantage for simulation and control of flotation circuits. King (1972) extended this further and applied multivariate distribution model for kinetic analysis of a flotation circuit. Huber-Fanu (1976) presented a general mathematical model taking into account the size distribution of feed material, the distribution of floatabilities in each size class and the mode of cell operation (batch or continuous).

2.6 KINETICS OF FLOTATION IN A CONTINUOUSLY OPERATED FLOTATION CELL

Most of the models discussed so far were developed for semi-batch flotation operation. A semi-batch flotation test does not, however, represent the actual continuous flotation cell, since the mineral and reagent concentration keep changing with time and steady state is never reached. The semi-batch testing was therefore criticized by Brown and Smith (1953) and they strongly recommended continuous flow testing for modelling and simulation. The merits and demerits of both semi-batch and continuous operations were discussed by Arbiter and Harris (1962), Woodburn et al (1970) and de Bryun and Modi (1956).

The recovery of a mineral in a continuously operated cell is governed not only by the distribution of rate constants, but also by the retention time of particles in the pulp

volume, which depends on the type of mixing or flow regime. The simplest case is the plug flow, wherein all the particles spend an equal amount of time in the cell before exit. From the aspect of residence time distribution or internal mixing of solids, a semi-batch flotation cell is equivalent to a theoretical continuous compartment with plug flow (Lynch et al, 1981). The other extreme condition is ideal mixing where complete homogeneity exists over the entire volume of the cell and, in such a cell, all the material has an equal probability of leaving the reactor. For a continuous cell with perfect mixing, the residence time distribution is given by exponential distribution (Levenspiel, 1962). Thus if $E(t)dt$ is the fraction of particles with a residence time of t to $t + dt$ in the cell, then Eq. 2.18 becomes

$$M_p(t) = \int_0^{\infty} \int_0^{\infty} M_p(K_a) E(t) \exp(-K_a t) dt dK_a \quad (2.22)$$

Many investigators have approximated the continuously operated sub-aeration cell to a perfect mixer (Woodburn and Loveday, 1966; Loveday, 1966; Woodburn et al, 1970; Jowett, 1961; Niemi and Paakkinen, 1969). However Niemi (1966a) observed that although liquid phase could be assumed to behave in a perfectly mixed flow, in the case of solid particles, particularly big particles, the assumption required modifications. He further added that even if solid particles were apparently perfectly mixed, their mean residence time differed from that of liquid phase.

In a significant contribution Woodburn et al (1971) and Mehrotra and Kapur (1975) showed that gamma distribution for $E(t)$ gave better results than exponential distribution. Mehrotra and Kapur (1975) applied distributed rate model for a continuously operated cell and suggested a method to estimate rate distribution from steady state data. Truly speaking, the plug flow and perfectly mixed flow are two ideal cases and these seldom represent the real systems. The total volume in real compartments often appears to behave as the summation of small volumes with perfect mixing, plug flow and stagnant (dead volume) characteristics. The effect which mixing characteristics may have on flotation behaviour due to changes in residence time distributions is quite significant (Lynch et al, 1981). Mehrotra and Saxena (1983) observed that in a continuously operated flotation cell, only a part of the cell volume behaves as a perfect mixer, the remaining being dead. This effective volume, which behaved as a perfect mixer varied with variations in operating conditions. Arbiter and Harris (1962) and Bull (1966) considered the flotation behaviour in a number of cells in series and have shown that if there are N perfectly mixed isolated compartments in series, and if the mean residence time τ per compartment is constant, then the fraction of floatable material in the N^{th} cell will be given as

$$M_{p_N} = (1 + K_a \tau)^{-N} \quad (2.23)$$

Jhonson (1972) considered back-mixing between the isolated industrial compartments. Thorne (1975, 1976) used the

mechanistic approach to model industrial sulphide flotation circuits. A lot of work is reported in literature on modelling of industrial flotation circuits, development of flotation circuit simulators, network synthesis etc. These being outside the scope of this study, are not included in this review.

2.7 EFFECT OF PROCESS VARIABLES ON FLOTATION KINETICS

2.7.1 Effect of Reagents

Both the structure of the collector molecules and its concentration affect the flotation rate by influencing the particle-bubble attachment rate. The effect of collector concentration on the flotation kinetics was studied by Beloglazov (1939), Schuhmann (1942) and Inoue and Imaizumi (1968). These studies showed that both recovery and flotation rate increased with the increase in collector concentration. However, this is achieved at the cost of selectivity. In order to maximise recovery, most industrial flotation circuits are operated at saturated collector dosage. But the effect of collector has to be studied only by experiments, no prediction can be made 'a priori'.

In the case of frother, Bennett et al (1958) showed that the increase in frother quantity caused increase in the number of bubbles with simultaneous reduction in the average size and enhancement of flotation rate resulted. Lynch (1981) attributed this increase in flotation rate to enhanced rate of water transfer from pulp to froth.

2.7.2 Effect of Aeration Rate

Many people reported that the overall flotation rate constant increased with increase in air-flow rate (Inoue and Imaizumi, 1968; Tewari and Biswas, 1969). Bennett et al (1958) pointed out that the enhanced aeration rate could lead to increased flotation rate only if the distribution of bubble size did not change, because the maximum flotation rate was achieved with larger number of smaller bubbles. Mehrotra and Kapur (1974) reported that flotation rate distribution function remained unaltered, but the argument of the function, the apparent rate constant got rescaled with change in the rate of aeration. Laplante et al (1983b) also observed that the overall flotation rate constant increased with increasing air-flow rate (AFR) and decreasing froth thickness. As AFR increased, the total bubble surface also increased, which caused the increase in flotation rate. However, increase in AFR also increased the overall bubble diameter, which caused a decrease in flotation rate. This resulted in the flotation rate constant passing through a maximum as AFR was increased.

2.7.3 Effect of Particle Size

It is very well known both by laboratory experiments as well as by industrial experience that particle size has a profound effect on the flotation behaviour of minerals. Mineral recovery is optimum only for a certain size range, and is decreased beyond this range. The effect of particle size has been studied very extensively and reviewed by Trahar and Warren (1976)

and Irahara (1981). Gaudin (1932) observed that selectivity was best for larger particles and decreased continuously with decreasing size. But even with larger particles the recovery was found to decrease. One reason for this decrease is that as the particles become larger, the density of the bubble-particle aggregate approaches that of the pulp, so the mineral-laden bubbles are less buoyant. This should, however, occur at much larger particle sizes than that at which the recovery starts decreasing (Jameson et al, 1977). Woodburn et al (1971) proposed a mechanism of particle detachment from the bubble, due to the hydrodynamic conditions prevailing in the cell. Attempts were made to explain the effect of particle size on recovery using the probabilities of collision, adhesion and detachment. In the case of very fine particles, the probability of collision is low because these have a tendency to follow the stream lines, and also due to their low momentum. With regard to the effect of particle size on flotation rate constant, Tomlinson and Fleming (1965) related the rate constant to the particle diameter in a simple way, as

$$K_a = qL^x \quad (2.24)$$

where q is a constant for a particular mineral. The exponent x is 2 for apatite, hematite and galena, and 1 for quartz, for a size range of 20-200 μm . Combining the probabilities of collision, adhesion and detachment, Woodburn et al (1971) proposed an expression for the overall rate constant K_a as a

function of particle size, and hydrodynamic parameter ω , as

$$K_a = \bar{K}(g) \frac{\omega^2}{L} \left[\exp\left(-\frac{\omega}{L^2}\right) \left[1 - \left(\frac{L}{L_{\max}}\right)^{1.5} \right] \right] \quad (2.25)$$

where $\bar{K}(g)$ is a function of the degree of hydration of particle of grade g , and it depends on the mineralogy of the particles and its surface treatment with the collector. Mehrotra and Kapur (1974, 1975) split the overall rate constant K_a as

$$K_a = \phi \eta(L) K^0 \quad (2.26)$$

where ϕ is an unspecified function of weighted mean bubble size and hydrodynamic conditions prevailing in the pulp and depends on the rate of aeration, $\eta(L)$ is a function of particle size only and K^0 depends on the particle characteristics such as grade and hydrophobicity. Changing only one of the variables, either aeration rate or particle size, they have attempted to solve the Eq. 2.26 using self-similar distribution in appropriately rescaled apparent flotation rate constants. Trahar (1981) observed that there exists a clear and fundamental relationship between the degree of hydrophobicity necessary to effect flotation and the particle size.

CHAPTER 3

MATERIALS AND METHODS

The details of the materials, experimental set-up and the methods used for this study are presented in this chapter and discussed.

3.1 MATERIALS

3.1.1 Characteristics of Ore Sample

The ore sample for the present study was received from the country's largest copper deposit at Malanjkhand, District Balaghat, Madhya Pradesh. The copper values in the ore are distributed in a number of sulphide and oxide minerals. The ore sample, labelled as 'medium oxide ore sample', was reported to contain 6-8 percent of total copper values in acid-soluble form.

Detailed petrographic analysis, (Table 3.1), indicated that the host rocks of ore mineralization and associated rocks were granites, aplites, metabasic rocks and quartz veins (Padmanabhan et al, 1986; Narasimhan and Rao, 1986). All these rock types showed hydrothermal alterations. The metabasic rocks have been extensively chloritized and were also occasionally veined by quartz. The sulphide mineralization was mostly confined to the quartz veins, though sporadic mineralization was observed in the adjoining granites and metabasics. It

could be seen from Table 3.1 that the sample contained less than 2 percent of copper values in the form of oxides (cuprite, tenorite and malachite). Chalcopyrite is the main copper bearing mineral accounting for 65 percent of metal values. Chalcocite and covellite are formed by secondary alteration of chalcopyrite and together account for 33 percent of the total copper values. The theoretical pure mineral grade for this ore sample was found to be 42.2 percent Cu.

Chemical analysis of carefully drawn representative samples gave 1.4 percent of total copper. Monitoring of beneficiation experiments was carried out by the assay of total copper only.

3.1.2 Chemical Reagents Used for Flotation

The flotation experiments were carried out using sodium isopropyl xanthate as collector and methyl isobutyl carbinol (MIBC) as frother. Sodium isopropyl xanthate was supplied by M/s Aceto Chemicals, Calcutta and the frother, MIBC, produced and supplied by M/s National Organic Chemicals Limited (NOCIL), Bombay, was obtained as a free sample from M/s Hindustan Zinc Limited.

The xanthate was added to the flotation pulp in the form of one percent solution and MIBC was added in drops from a dropper.

For carrying out flotation experiments at alkaline pH, sodium carbonate (AR) and sodium hydroxide (GR) solution were used as pH modifier.

TABLE 3.1
Mineralogical Composition of the Ore Sample

Mineral	Weight, Percent	Distribution of Cu, Percent
Quartz and Feldspars	93.67	
Ferromagnesian Minerals (Chlorite, Biotite etc.)	1.52	
Other Transparent Minerals (Apatite, Calcite, Topaz etc.)	0.02	
Opagues		
Chalcopyrite	3.03	65.1
Chalcocite	0.55	27.8
Covellite	0.13	5.3
Cuprite	0.02	1.1
Malachite	0.02	0.7
Pyrite	0.58	
Magnetite and Other Iron Oxides	0.46	
Total	100.00	100.0

3.2 EXPERIMENTAL SET-UP

3.2.1 Batch Ball Mill

All the grinding were carried out in Denver Laboratory batch ball mill with a ball charge of 20 kg occupying about 40 percent of the inside mill volume. The mill revolved at about 85-90 rpm. The feed material along with the required amount of water was fed through the feed opening of the mill and it was run for predetermined duration of time after closing with its lid. After grinding, the material was discharged along with the balls and was passed through 35 mesh sieve after removing the balls. The finer fraction was taken for flotation experiments.

3.2.2 Leeds Open Top Flotation Cell

Flotation experiments were carried out in Leeds open top flotation cell. This cell with closed and open tops, was developed by C.C. Dell (Dell and Bunyard, 1972; Dell and Hall, 1981), at the University of Leeds, U.K., and has a number of advantages over the conventional flotation cells. It is possible with this cell to achieve a high degree of reproducibility of test results (Apling and Ersayin, 1986), and this accuracy is obtained by an excellent control of four important variables, (i) impeller speed, (ii) air flow rate, (iii) pulp level and (iv) froth removal.

The flotation set-up is shown in Figure 3.1. This cell essentially consists of a plexiglass chamber of about 3 litre capacity mounted on a base. Impeller shaft enters the chamber

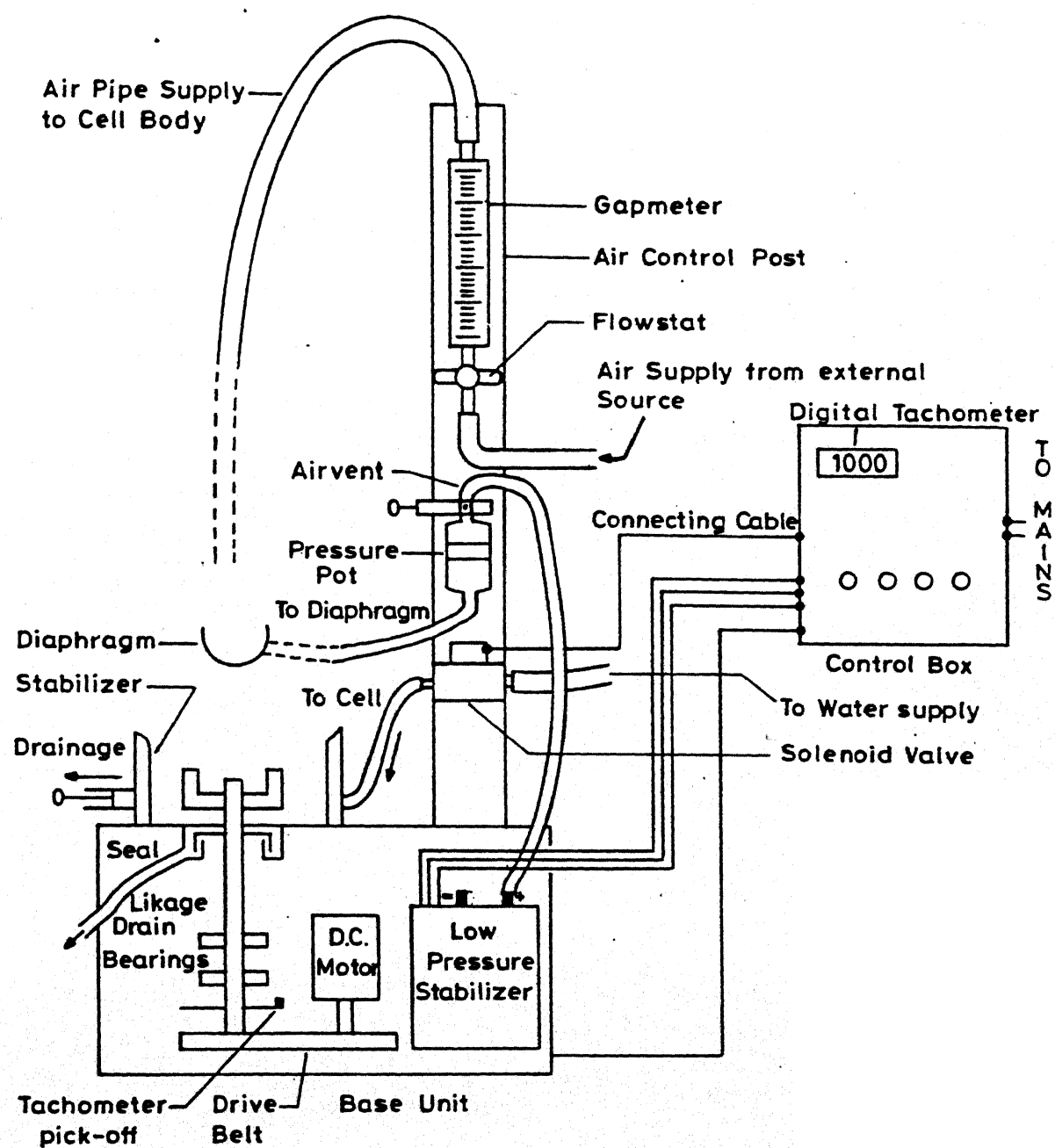


FIGURE 3.1 Schematic Diagram of Leeds Open Top Flotation Cell.

from the bottom through proper seals and drain water traps. The impeller is a circular plate with vertical fingers fixed along the circumference. Air is discharged through a separate tube near the impeller fingers, where they are finely divided and dispersed. Air flow rate is controlled by a floatat controller and measured by a "Platon" gapmeter. The pulp level is maintained constant by addition of reagent water from an overhead reservoir, and water addition is controlled by a solenoid valve activated by a pressure diaphragm at the side of the cell body, a pressure pot and low pressure stabiliser unit. The impeller speed is accurately monitored with a thyristor controller and a digital tacho display. The froth removal is facilitated by the open-top design, which provides an unrestricted access to the cell top. The froth depth can be increased by increasing the height of the cell with plexi-glass sections. However, there was a recurring problem of the shaft seal at the bottom of the cell getting worn out leading to leakage of slurry from the bottom.

3.3 EXPERIMENTAL TECHNIQUE

3.3.1 Feed Preparation

The ore sample when received was distributed in the size range from 50-60 mm pieces to very fine particles. The whole sample was stage crushed in jaw and roll crushers to get a product all passing through 6 mm screen. For the purpose of future reference a representative sample was drawn from this

crushed material by coning and quartering. The remaining material was stored in a container for use as the feed for all batch experiments.

The general strategy adopted for the preparation of feed for flotation experiments is mainly as follows: One kg of the crushed material was ground in the laboratory batch ball mill, described in Section 3.2.1, at a pulp density of 67 per cent solids (water - 500 ml) for a specified duration of time. Since the mill product contained a few coarse unground particles, which may jam the impeller in the Leeds cell, these were removed by passing the mill product through 35 mesh sieve. The finer fraction was floated in Leeds cell. Figure 3.2 gives the details of the experimental procedure.

3.3.2 Semi-batch Flotation Experiments

The mill product was allowed to settle and after decanting the supernatant water, it was transferred into the Leeds cell. A pulp sample was drawn after thoroughly suspending the material for estimating the particle size distribution of the flotation feed. Required amount of sodium isopropyl xanthate was added and conditioning was carried out for 4 minutes. Then MIBC was added in drops and the conditioning was continued for additional one minute before aeration was started. At the end of the five minutes air was let in at a specified rate and froth collection was started after giving a 15 sec. froth build up time. In general flotation was carried out for a total time

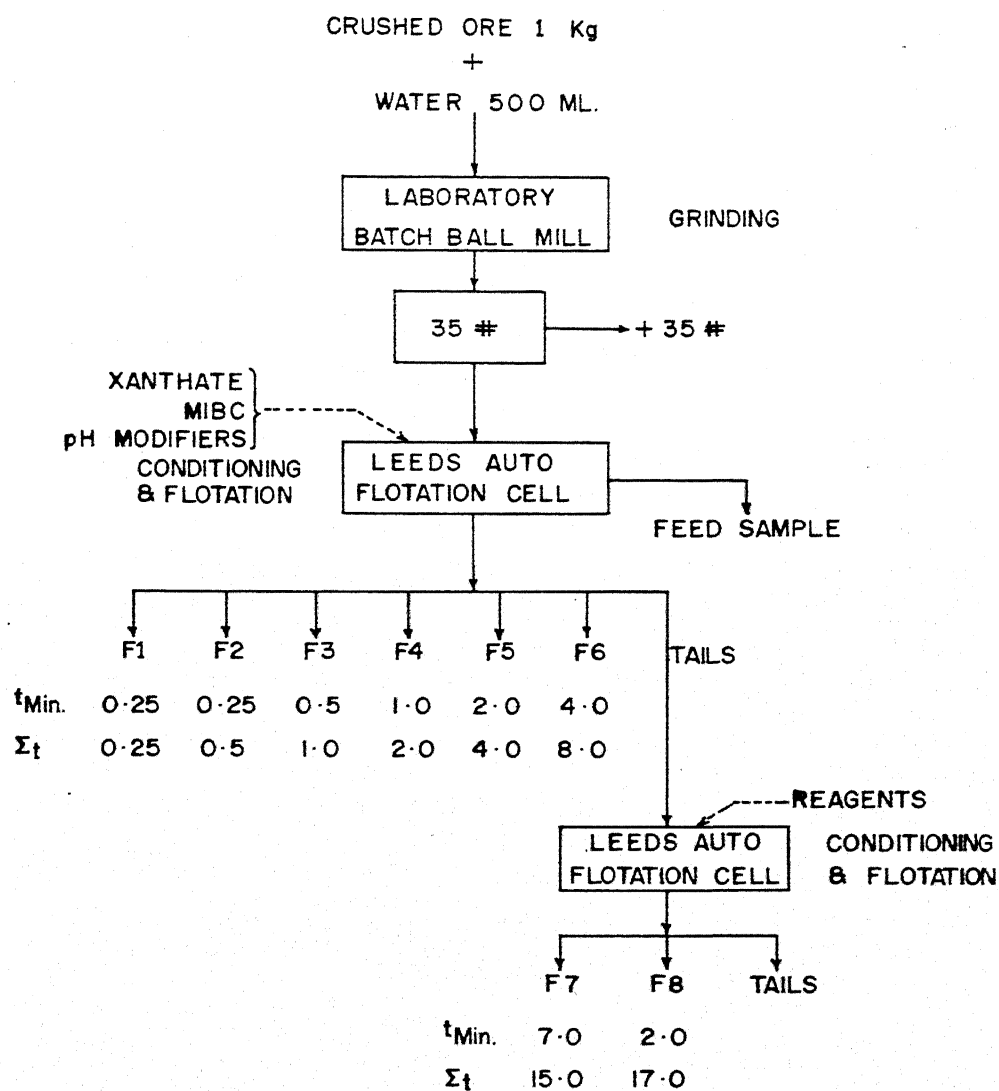


FIGURE 3.2 General Scheme of Batch Experiments.

of 17 minutes, with the addition of reagents and conditioning once again after initial 8 minutes of flotation. The pulp level was maintained constant by the addition of reagent water from an overhead reservoir through the pulp level control-valve mechanism of the Leeds cell.

The float was collected on incremental time basis to generate kinetic data. From the starting time of froth collection, the float was collected separately at the cumulative times of 0.25, 0.5, 1.0, 2.0, 4.0, 8.0, 15.0 and 17.0 minutes (see Figure 3.2) and the float fractions were termed F1 to F8 respectively. All the float fractions were dried, collected and weighed. Later these were sampled by coning and quartering after breaking the lumps and these samples were chemically analysed for total copper content using iodimetric method. A summary of batch test conditions and variables studied is shown in Table 3.2. The experiments are listed with respect to the variable studied.

TABLE 3.2
Summary of Experimental Conditions

Expt. No.	Grinding Time min	Xanth. ml	MIBC Drops	pH	RPM	Airflow Rate lpm	Remarks
2	5	7	3	7	700	5	Replicate
4	5	7	3	7	700	5	
24	5	7	3	7	700	5	
19	3	7	3	7	700	5	Grinding Time
8	8	7	3	7	700	5	
1	10	7	3	7	700	5	
12	5	4	3	7	700	5	Xanthate
13	5	9	3	7	700	5	
14	5	12	3	7	700	5	
15	5	7	5	7	700	5	Frother
5	5	7	5	9.5	700	5	pH
6	5	7	5	10.5	700	5	
17	5	7	5	7	900	5	RPM
27	5	7	5	7	1000	5	
16	5	7	5	7	700	7	Airflow Rate
21	5	7	5	7	700	10	
29A	5	7	5	7	700	5	-35+100
29B	5	7	5	7	700	5	-100+200
29C	5	7	5	7	700	5	-200
25	5	7	5	7	700	5	Rougher 1 and 2 + Scavenger
26	5	7	5	7	700	5	Cleaner

CHAPTER 4

RESULTS AND DISCUSSION

The results of semi-batch flotation experiments conducted on Malanjkhand copper ore using Leeds open top flotation cell, with various process and equipment conditions are presented, analysed and discussed in this chapter. Initially, the general method adopted to analyse the experimental results is described, followed by the results and discussion. An alternate transformation for use in the data analysis is also described.

4.1 EXPERIMENTAL RESULTS

The batch experiments carried out as described in the previous chapter yielded the weight in grams of various product fractions and their corresponding copper assays. From this raw data, the material balance was computed in the following manner.

The weight percent of i^{th} product fraction is given as

$$w_i \% = \frac{w_i}{\sum_{i=1}^n w_i} \times 100 \quad (4.1)$$

where w_i is the weight in grams of i^{th} product fraction and n is the total number of product fractions. The cumulative weight percent w_i is given as

$$w_i \% = \frac{\sum_{j=1}^i w_j}{\sum_{j=1}^n w_j} \times 100 \quad (4.2)$$

The percent copper recovery d_i of i^{th} product fraction is given as

$$d_i \% = \frac{w_i g_i}{\sum_{i=1}^n w_i g_i} \times 100 \quad (4.3)$$

where g_i is the copper assay of the i^{th} product fraction. The cumulative percent copper recovery, D_i is calculated as

$$D_i \% = \frac{\sum_{j=1}^i w_j g_j}{\sum_{j=1}^n w_j g_j} \times 100 \quad (4.4)$$

The cumulative grade G_i is computed using the formula

$$G_i \% = \frac{\sum_{j=1}^i w_j g_j}{\sum_{j=1}^n w_j} \times 100 \quad (4.5)$$

The raw data and the derived values of a typical experiment are shown in Table 4.1. For graphical presentation of experimental results, the cumulative figures are generally used.

4.2 ANALYSIS OF EXPERIMENTAL DATA

Since the feed for all the batch experiments was taken from the ore-stock stored in a container, there was a slight variation in the back calculated copper assay of the feed from

TABLE 4.1

Results of a Typical Batch Experiment

Product Fractions	Flotation Time t min	Cum. Time Σt min	Raw Data		Derived Data				
			w_1 gm	g_1 Cu	w_1 %	W_1 %	G_1 %	d_1 %	D_1 %
Float 1	0.25	0.25	17.1	21.50	2.4	2.4	21.50	30.0	30.0
Float 2	0.25	0.50	6.9	18.19	1.0	3.4	20.53	10.6	40.6
Float 3	0.50	1.0	4.2	13.59	0.6	4.0	19.49	4.7	45.3
Float 4	1.0	2.0	10.5	12.64	1.5	5.5	17.62	11.0	56.3
Float 5	2.0	4.0	12.5	6.56	1.8	7.3	14.89	6.9	63.2
Float 6	4.0	8.0	29.3	4.02	4.2	11.5	10.92	9.8	73.0
Float 7	7.0	15.0	54.3	4.02	7.8	19.3	8.13	18.3	91.3
Float 8	2.0	17.0	10.2	1.83	1.5	20.8	7.68	1.6	92.9
TAIL			553.0	0.154	79.2	100.0	1.719	7.1	100.0
Total			698.0		100.0				

experiment to experiment. To nullify the effect of this variation on flotation behaviour, the w_i were normalised with respect to the feed grade. This normalised concentrate weight, termed as 'unit weight' is defined as

$$\text{Unit weight} = \frac{w_i}{\sum_{i=1}^n w_i g_i} \quad (4.6)$$

Unit weight thus represents the weight of concentrate that would be obtained if the feed grade were 1 percent metal.

The results were analysed using a distributed rate-constant kinetic model. As mentioned in Chapter 2, the total material remaining in the pulp at time t can be written as (cf. Eq. 2.18)

$$M_p(t) = \int_0^{\infty} M_p(K_a) \exp(-K_a t) dK_a \quad (4.7)$$

where $M_p(K_a) dK_a$ is the mass fraction of particles with apparent overall rate constant in the range K_a to $K_a + dK_a$. The objective is to estimate the $M_p(K_a)$ from the experimental data, $M_p(t)$, at discrete flotation time intervals.

As it has already been established (Harris and Chakravarty, 1970; Kapur and Mehrotra, 1974) that $M_p(t)$ in Eq. 4.7 is nothing but the Laplace transform of $M_p(K_a)$ and therefore $M_p(K_a)$ is the inverse of the Laplace transform. In the case of discrete experimental data $M_p(t)$, this inverse must be computed by numerical means. But the numerical inversion of Laplace transform is not an easy task and the problems

associated with this inversion have been highlighted elsewhere (Kapur and Mehrotra, 1974). Since the inverse of Laplace transform is a highly unstable operator, even slight errors in the experimental data give erroneous results if conventional methods of inversion are used. So a numerical method developed by Kapur and Mehrotra (1974), which can tolerate reasonable amount of errors in the experimental data, was used to estimate the rate constant distribution. Details of this method are given in Appendix A1.

One of the important features of this technique is that the rate distribution is evaluated in the cumulative mode. Let $R(K_a)$ be the cumulative distribution function, such that

$$M_p(K_a) = - \frac{dR(K_a)}{dK_a} \quad (4.8)$$

Substitution of Eq. 4.8 in Eq. 4.7 followed by integration by parts gives

$$\bar{M}_p(t) = \int_0^{\infty} R(K_a) \exp(-K_a t) dK_a \quad (4.9)$$

$$\text{where } \bar{M}_p(t) = [1 - M_p(t)]/t \quad (4.10)$$

A computer program was developed to evaluate $R(K_a)$ using the technique described in Appendix A1. The inputs to the program were the experimental recovery values (D_i) as a function of time. Since the efficiency of the inversion technique is good only when a large number of input data points are used, the additional data points were generated by plotting D_i against t

and reading out D_i values at the intervals of 0.25 minutes from the plot. In order to establish the validity of this technique, $R(K_a)$ values, estimated by the inversion technique, were used to back calculate the D_i values. In all the cases, a very good fit was obtained. Table 4.2 gives the typical results of one of the experiments.

4.3 RESULTS AND DISCUSSION

4.3.1 Reproducibility of Test Results

Prior to carrying out the actual experiments to study the effect of different control variables on the flotation performance, replicate experiments were performed using Leeds cell to determine the reproducibility of test results. Some typical results of these experiments are shown in table 4.3. A reasonably close agreement was obtained indicating a good reproducibility comparable to reported results (Liddell and Dunne, 1984; Apling and Ersayin, 1986). Comparative studies on the reproducibility of test results obtained from Leeds cell and conventional Denver cell have been carried out in this laboratory (Mehrotra and Singh, 1987), which indicate that a better reproducibility is obtained with Leeds cell. Good reproducibility of experimental results in our case is attributed to the positive control of airflow rate, impeller speed, pulp level and finally to the unrestricted access to the cell top in Leeds open-top cell. The standard deviations were found to be smaller than those reported for Leeds 'Autofloat'

TABLE 4.2

Comparison between Experimental and
Calculated Values of $M_p(t)$

Expt. No. 24

Time, min	$M_p(t)$ exptl.	$M_p(t)$ model
0.0	1.0	1.0
0.25	0.700	0.700
0.5	0.594	0.596
1.0	0.530	0.520
2.0	0.437	0.452
4.0	0.368	0.370
8.0	0.270	0.274

$$\chi^2 = \left[\frac{M(t)_{\text{exptl}} - M(t)_{\text{model}}}{M(t)_{\text{exptl}}} \right]^2 = 0.0054$$

TABLE 4.3
Reproducibility of Batch Tests

Float Fraction	Cum. Time min	Cum. Unit Weight					Cum. Copper Recovery, %				
		Expt. 2	Expt. 4	Expt. 24	Mean	σ_{n-1}	Expt. 2	Expt. 4	Expt. 24	Mean	σ_{n-1}
F1	0.25	1.2	1.3	1.4	1.3	0.10	29.8	28.9	30.0	29.6	0.6
F2	0.5	1.7	1.8	2.0	1.8	0.15	38.7	37.8	40.6	39.0	1.4
F3	1.0	2.2	2.4	2.3	2.3	0.10	44.7	44.8	45.3	44.9	0.3
F4	2.0	2.9	3.3	3.2	3.1	0.21	51.5	52.1	56.3	53.3	2.6
F5	4.0	4.0	4.9	4.2	4.4	0.47	58.4	61.3	63.2	61.0	2.4
F6	8.0	6.2	7.7	6.6	6.8	0.78	70.1	70.9	73.0	71.3	1.5

Note: For experimental conditions, see TABLE 3.2.

closed-top flotation cell, indicating the supremacy of open-top design.

4.3.2 Effect of Grinding Time

Experiments were carried out to determine the effect of grinding time on the flotation behaviour of copper minerals. Grinding was performed in the laboratory batch ball mill for 3, 5, 8 and 10 minutes and the flotation of the ground material was carried out as described in Section 3.2. The particle size distribution of the feed, ground for different durations of time are given in Table 4.4. When the grinding was increased from 3 to 10 minutes, the amount of -200 mesh material in the flotation feed increased from 47.5 to 81.1 percent. The material ground for 5 minutes had about 24 percent of it passing through 325 mesh sieve. The results of experiments carried out at different grinding times are presented in Table 4.5. The copper recovery values are plotted in Figure 4.1 against flotation time under different conditions of grinding time. It can be seen that the flotation behaviour is far superior in the case of material ground for 3 minutes, where the amount of -200 mesh material is only 47.5 percent. But as it increased due to grinding for longer durations of time, the weight collected (unit weight) and the copper recovery both decreased considerably indicating that the fines did not float well. The experimental data was analysed using distributed rate constant kinetic model and the flotation rate distributions were estimated as described in Section 4.2. The cumulative rate

TABLE 4.4

Particle Size Distribution of Ground Ore

Grinding Time mesh	Cumulative Weight Percent Finer			
	3 min	5 min	8 min	10 min
35	100.0	100.0	100.0	100.0
65	89.2	96.3	98.8	98.8
100	77.1	90.3	95.9	96.3
150	65.0	86.1	90.0	89.9
200	47.5	67.5	72.5	81.1
240	-	66.0	-	-
300	-	54.6	-	-
325	-	24.3	-	-

TABLE 4.5

Effect of Grinding Time on Flotation Behaviour

Float Fraction	Cum. time min	Cum. Unit Weight			Cum. Copper Recovery, %			
		Expt. 19	Expt. 4	Expt. 18	Expt. 19	Expt. 4	Expt. 18	Expt. 1
F1	0.25	1.7	1.3	1.0	43.3	28.9	21.8	22.4
F2	0.5	2.5	1.8	1.4	58.4	37.8	29.4	31.0
F3	1.0	3.4	2.4	2.2	67.5	44.8	41.5	40.1
F4	2.0	4.4	3.3	2.9	73.8	52.1	50.2	47.8
F5	4.0	5.8	4.9	4.5	79.4	61.3	57.8	52.8
F6	8.0	7.4	7.7	8.1	83.9	70.9	68.5	59.2
Grinding Time, mm	3		5	8	10	5	8	10
-200 Mesh,	47.5	67.0	72.5	81.1	47.5	67.0	72.5	81.1

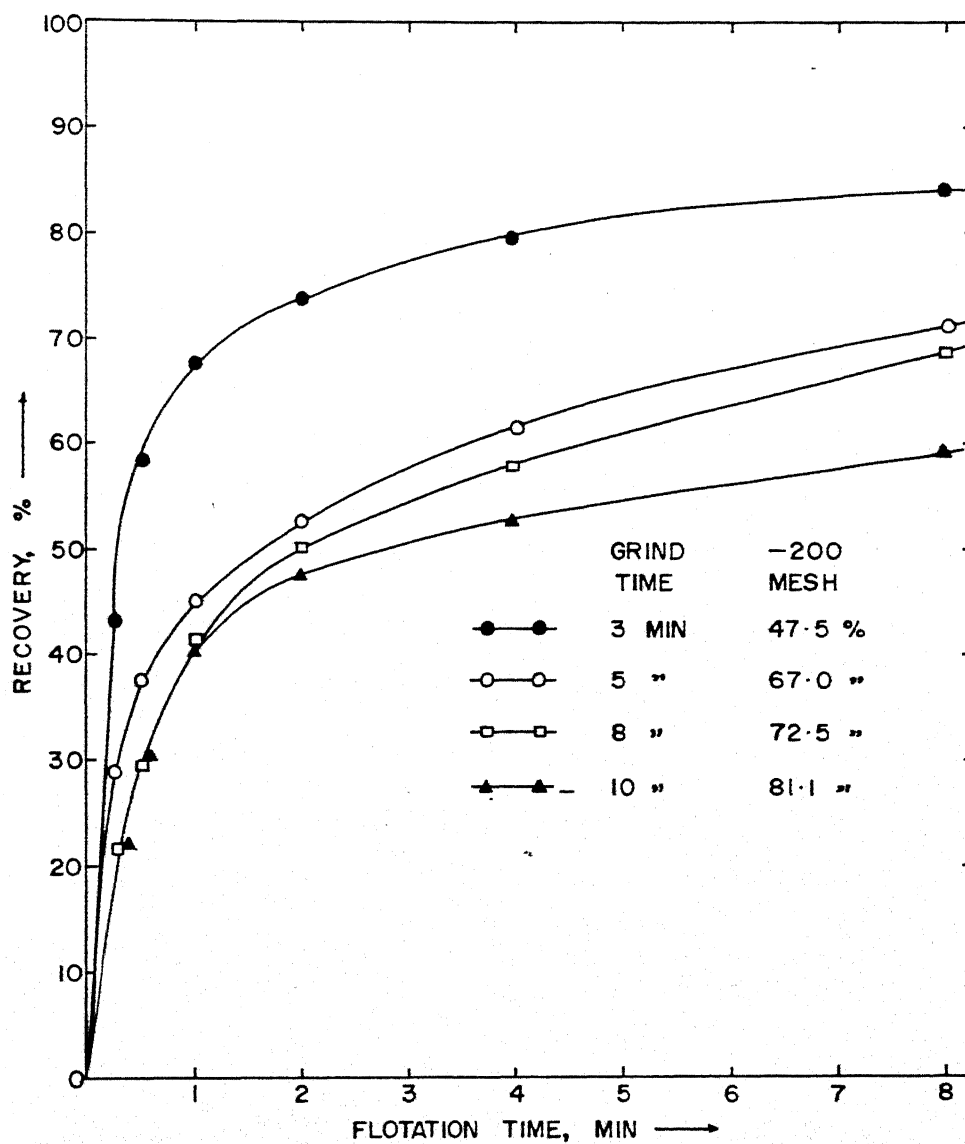


FIGURE 4.1 Effect of Grinding Time on Flotation Recovery (Xanthate 7 ml; MIBC 3 drops; rpm 700; pH 7; Air flow 5 lpm).

distributions are plotted in Figure 4.2. The frequency rate distributions, evaluated by numerical differentiation of $R(K_a)$, are shown in Figure 4.3. Though numerical differentiation is in general less accurate, a qualitative idea can nevertheless be obtained as to how the frequency rate distribution changes with grinding time. It can be seen that the maximum flotation rate, K_{am} decreases as grinding time is increased. In addition, the area under the peak increases indicating that the fraction of particles with low rate constants increases with the grinding time, thus explaining why flotation behaviour gets deteriorated as grinding time is increased. This phenomenon is indicated in Figure 4.2 also, where the median flotation rate $K_a(0.5)$, i.e. the flotation rate at which $R(K_a)$ is 0.5, also decreases drastically with increasing grinding time.

It was reported earlier (Padmanabhan et al, 1986) that liberation of valuable minerals in this ore was complete even at a coarser grinding. Any further grinding only led to the preferential grinding of the softer copper minerals in comparison to the very hard quartz host rocks, and eventual loss of copper recovery in the subsequent flotation stage.

The above experiments necessitated an inquiry into the dependence of flotation behaviour on particle size which was carried out and is described in the next section.

4.3.3 Effect of Particle Size

Flotation experiments were carried out on different sized fractions of ground material to find out the effect of

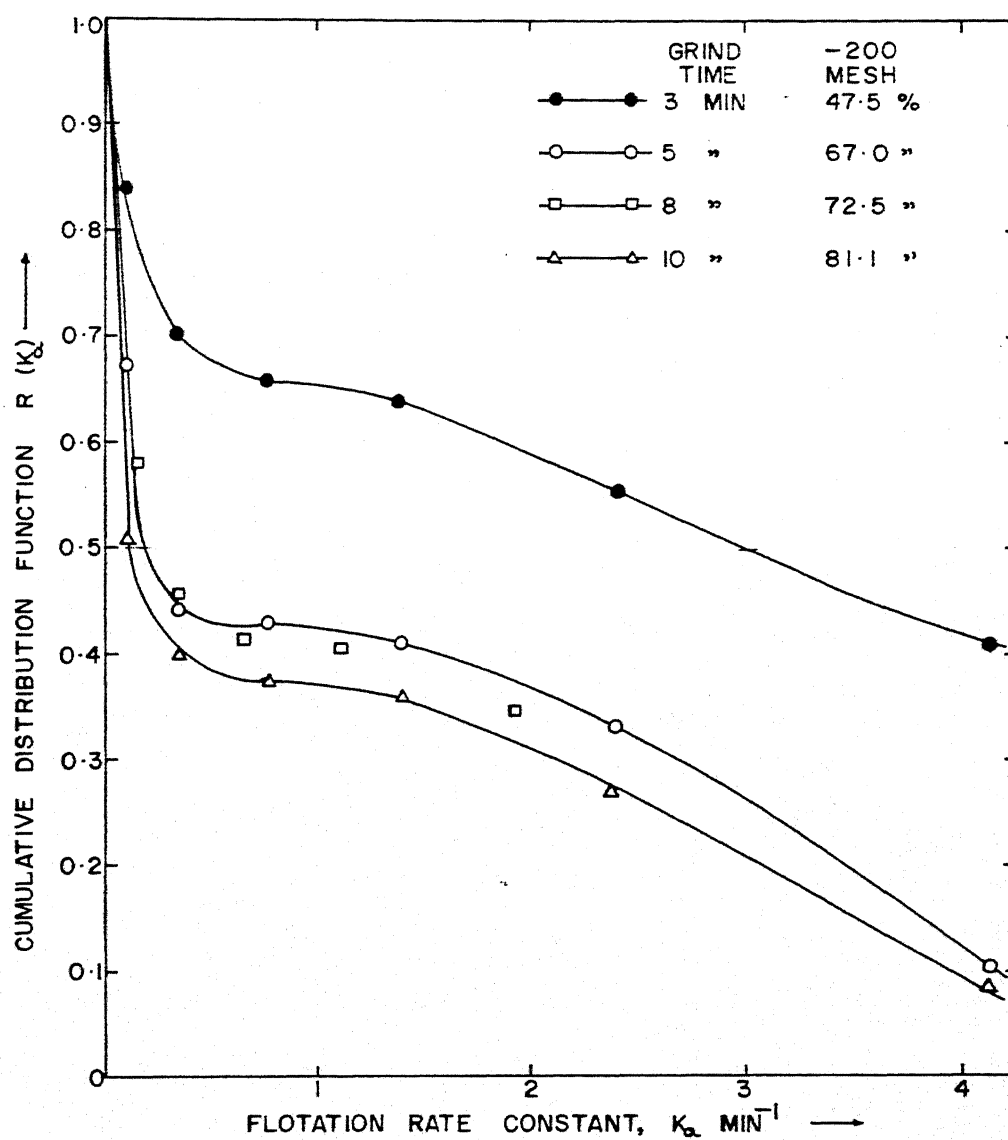


FIGURE 4.2 Effect of Grinding Time on Cumulative Apparent Flotation Rate Constant Distribution.

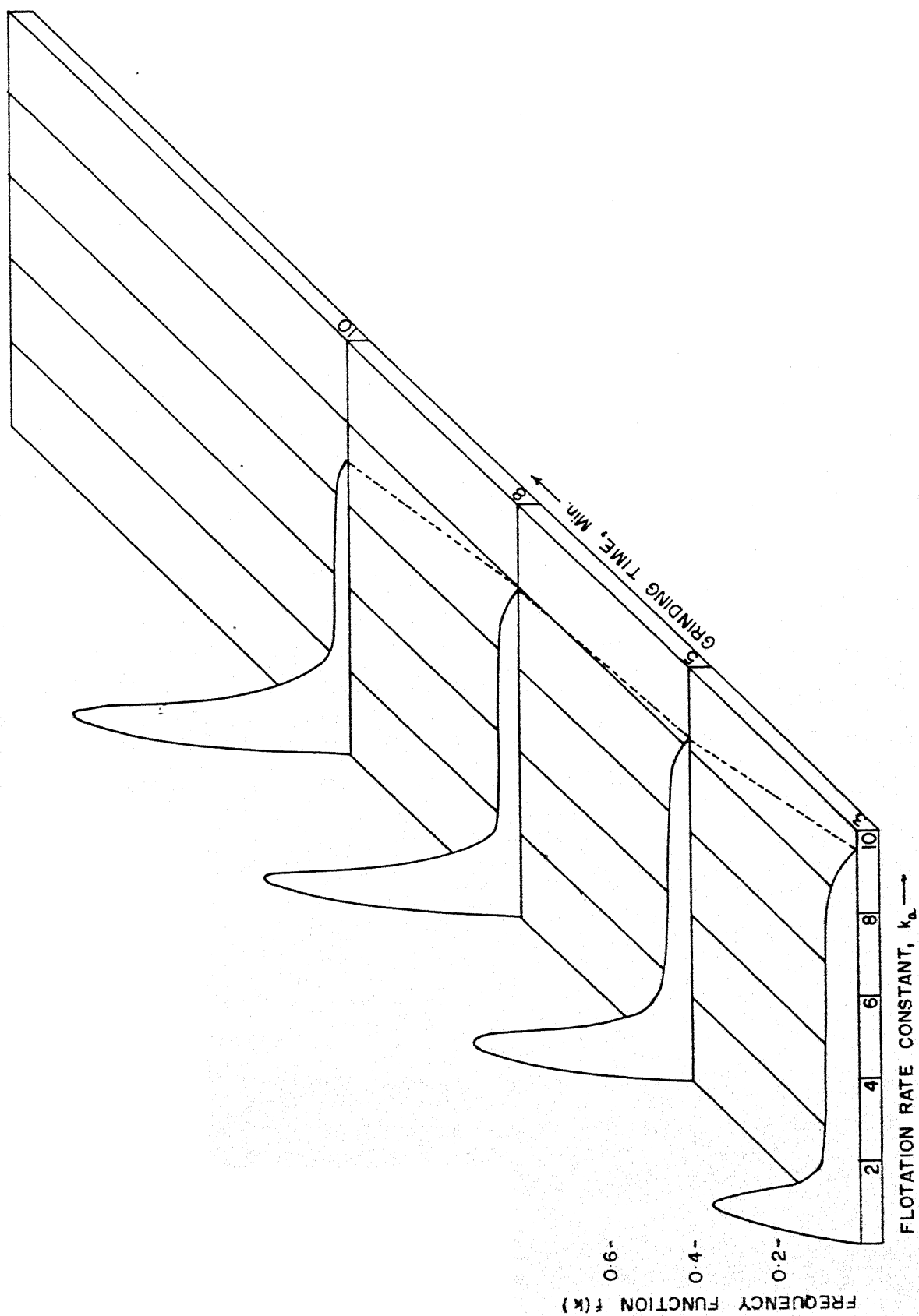


FIGURE 4.3 Effect of Grinding Time on Apparent Flotation Rate Constant Distribution (frequency diagram).

particle size on flotation behaviour. The grinding was done for 5 minutes and adequate number of batches of crushed ore were ground in order to get sufficient material in each size fraction to maintain the same pulp density during flotation. The ground ore was classified into three size fractions, namely -35+100, -100+200 and -200 mesh, and these were separately floated under the same conditions (cf. Table 3.2, Expt. 29). The experimental results are given in Table 4.6 and the unit weight is plotted against flotation time in Figure 4.4 for different particle size fractions. It may be seen that the unit weight of the float increased drastically as the size became finer. The recovery-flotation time plots shown in Figure 4.5 indicate that the copper recovery increased when the particle size was changed from -35+100 to -100+200 mesh, but decreased on going to finer size, -200 mesh. The same information is obtained from Figure 4.6 wherein recovery is plotted as a function of particle size. All these indicate that as the particle size was reduced, more material started floating by entrainment, as shown by increase in concentrate weight, and selectivity was getting lost as shown by the decrease in recovery from -200 mesh material. The distribution of copper values in various size fractions and the amount recovered from each size are shown in the bar-chart, Figure 4.7. The recovery of copper from the -100+200 mesh material was almost 100 percent while it was 87 percent from -35+100 mesh size. On the other hand, it was only about 70 percent from -200 mesh size, wherein 75 percent of copper values occur.

TABLE 4.6

Effect of Particle Size on Flotation Behaviour

Float Fraction	Cum. Time min	Cum. Unit Weight			Cum. Copper Recovery, %		
		Expt. 29A	Expt. 29B	Expt. 29C	Expt. 29A	Expt. 29B	Expt. 29C
F1	0.25	1.9	2.6	1.8	50.6	75.5	30.8
F2	0.5	3.1	3.4	2.9	79.4	86.0	40.9
F3	1.0	3.5	4.2	3.9	83.7	91.9	47.7
F4	2.0	3.8	4.8	5.5	85.3	94.4	56.0
F5	4.0	3.9	5.5	7.3	86.0	96.6	62.9
F6	8.0	4.2	6.1	9.7	86.9	98.1	69.3
Particle Size		-35+100	-100+200	-200	-35+100	-100+200	-200

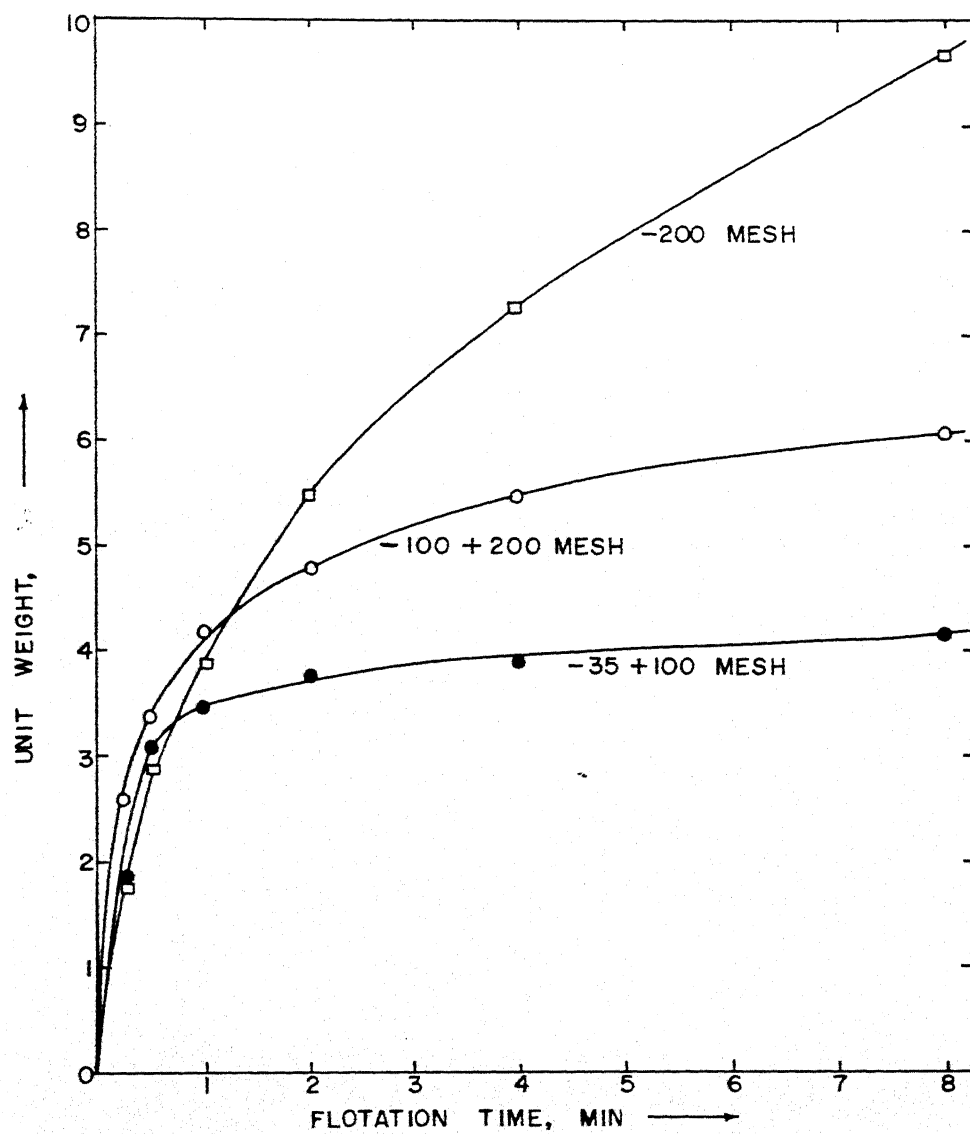


FIGURE 4.4 Effect of Particle Size on Weight Floated.

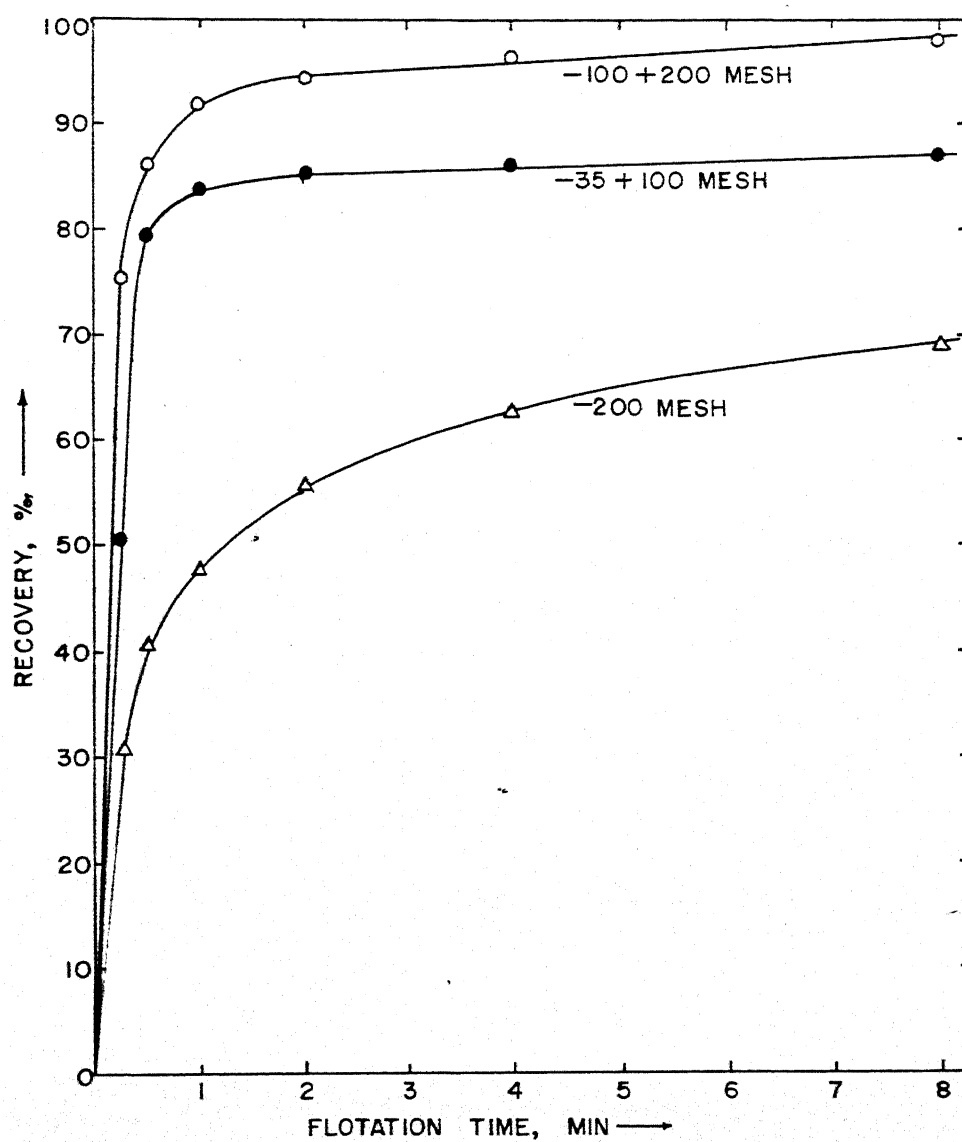


FIGURE 4.5 Effect of Particle Size on Flotation Recovery (Xanthate 7 ml; MIBC 3 drops; rpm 700; pH 7; air flow 5 lpm).

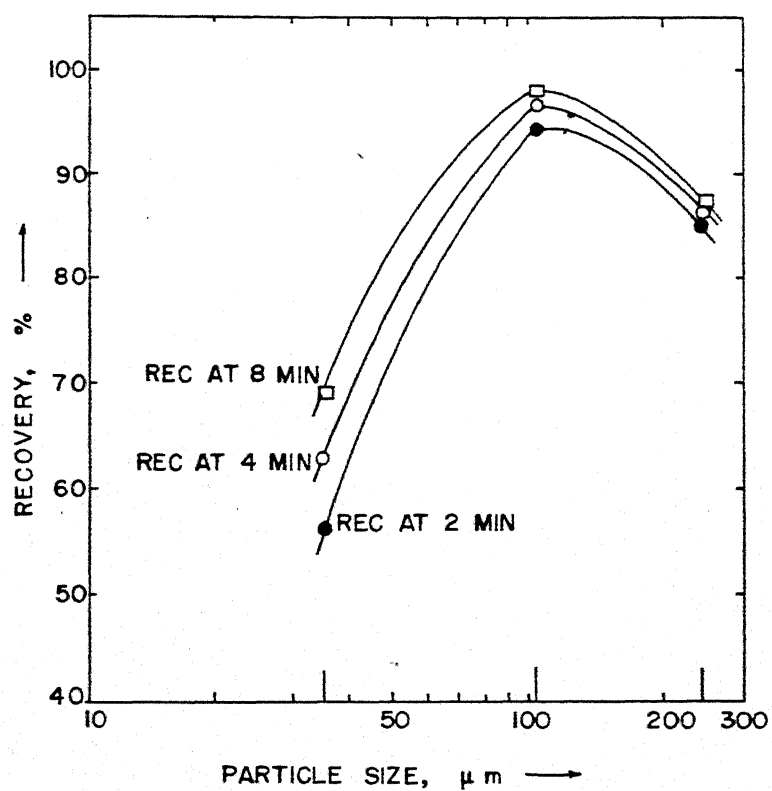


FIGURE 4.6 Effect of Particle Size (recovery-particle size plot).

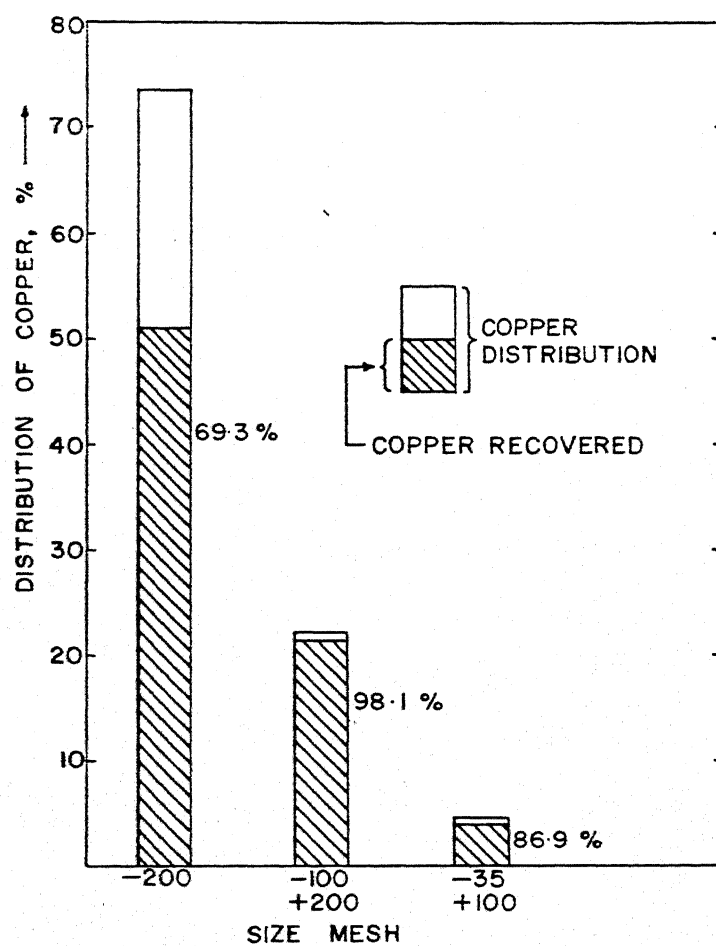


FIGURE 4.7 Bar-chart Showing Distribution and Recovery of Copper in Different Size Fractions.

Although percent recovery was very good in coarser sizes, the copper distribution was much less than that in -200 mesh fraction. So this vividly explains the flotation behaviour that was observed in the previous section.

It may be seen that the -100+200 size fraction, from which the copper recovery is extremely good, gradually decreased from about 30 to 15 percent (Table 4.4), when grinding time was increased from 3 to 10 minutes. Similarly the amount of -35+100 size fraction, from which the recovery is still very good, decreased rapidly from 23 to 3 percent with increase in grinding time. On the other hand the amount of -200 mesh material from which the recovery is rather less, increased from 47.5 to 81.1 percent with increase in grinding time. Reduction in the amounts of -35+100 and -100+200 mesh size fractions, from which the recovery is good, and the increase in the amount of -200 mesh material from which the recovery is poor, both explain the experimental observation in which recovery decreased with grinding time (Figure 4.1) much against the common belief that increased grinding produced more liberation and hence, higher recovery.

It can also be seen that when grinding time was increased from 3 to 5 minutes the amount of -200 mesh material increased from 47.5 to 67.5 percent (increase by 20 percent), but from 5 to 8 minutes, it did not increase much (only by 5 percent) and again from 8 to 10 minutes the increase in the production of -200 mesh material was about 9 percent. This phenomena is reflected in flotation behaviour, as shown in

Figures 4.1 and 4.2. The recovery and the rate distribution curves for 5 and 8 minutes are lying very close.

In this case also, kinetic analysis was done and the flotation rate distributions estimated (Figure 4.8) showed that -100+200 mesh material had higher amount of fast floating species, as evidenced by the highest value of $K_a(0.5)$ for this size fraction. For both the coarser and finer fractions, the $K_a(0.5)$ values were less. Half of the -200 mesh material had the apparent flotation rate constant less than about 0.5 (Figure 4.8). Mehrotra and Kapur (1974) observed that the plot of $K_a(0.5)$ versus particle size exhibited a characteristic peak in the intermediate size range and this present study confirmed it.

4.3.4 Effect of pH

Since the flotation behaviour of various minerals is affected by pulp pH, experiments were conducted on the ore sample at three different pH conditions. In all these experiments the grinding was done for 5 minutes. The natural pH of the pulp without the addition of any modifier was found to be 7. Sodium carbonate (AR) and sodium hydroxide (GR) were used to get pH values of 9.5 and 10.5 respectively. The results of these experiments are presented in Table 4.7. The recovery-time plots are shown in Figure 4.9 for the three different pH conditions. It could be seen that the pH has a profound effect on the copper flotation. There was an improvement in recovery with increase in pH. There was no significant increase in the

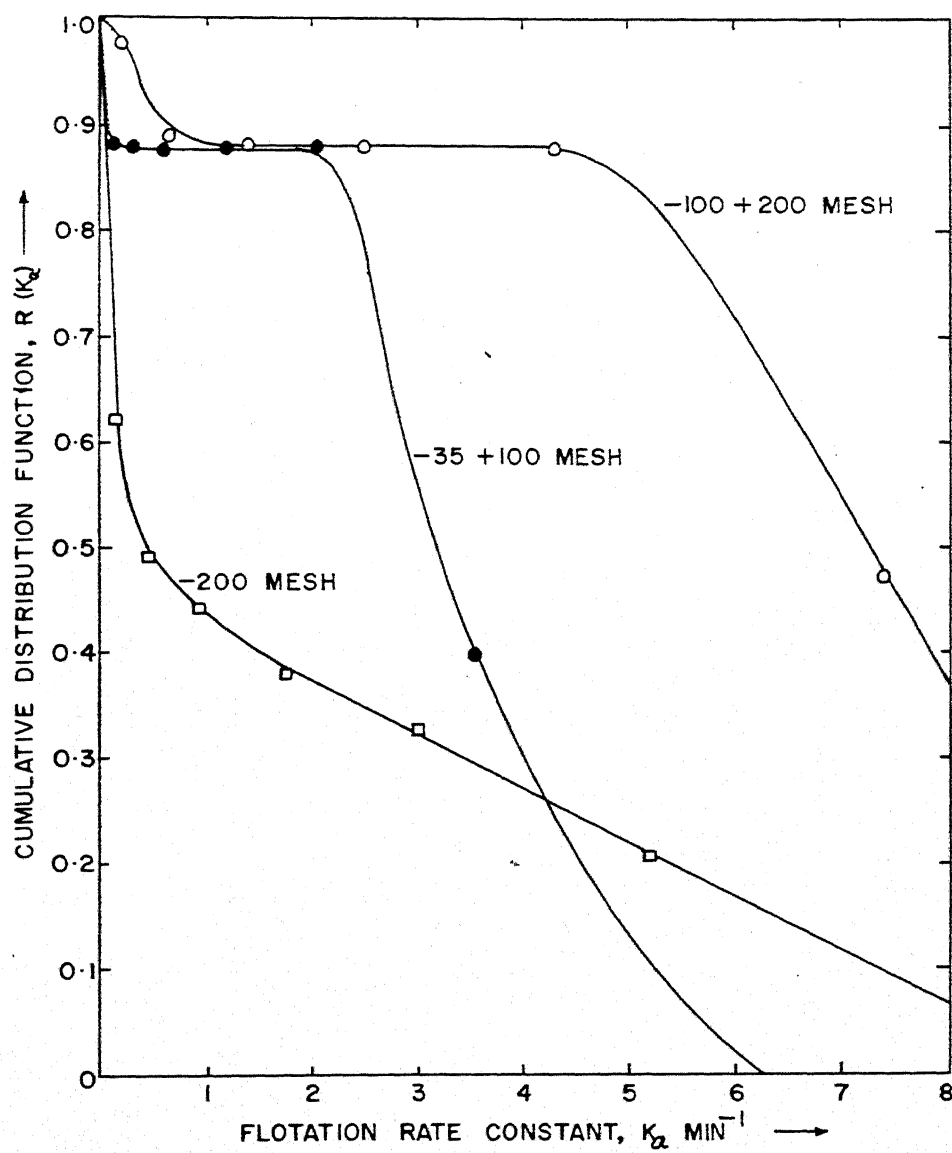


FIGURE 4.8 Effect of Particle Size on Cumulative Apparent Flotation Rate Constant Distribution.

TABLE 4.7
Effect of pH on Flotation Behaviour

Float Fraction	Cum. Time min	Cum. Unit Weight			Cum. Copper Recovery, %		
		Expt. 4	Expt. 5	Expt. 6	Expt. 4	Expt. 5	Expt. 6
F1	0.25	1.3	1.8	1.8	28.9	45.3	46.6
F2	0.5	1.8	2.4	2.4	37.8	56.1	57.1
F3	1.0	2.4	3.2	3.5	44.8	67.8	71.6
F4	2.0	3.3	4.1	4.4	52.1	74.7	80.0
F5	4.0	4.9	5.4	5.6	61.3	80.9	85.4
F6	8.0	7.7	7.5	7.5	70.9	85.6	90.0
pH		7	9.5	10.5	7	9.5	10.5

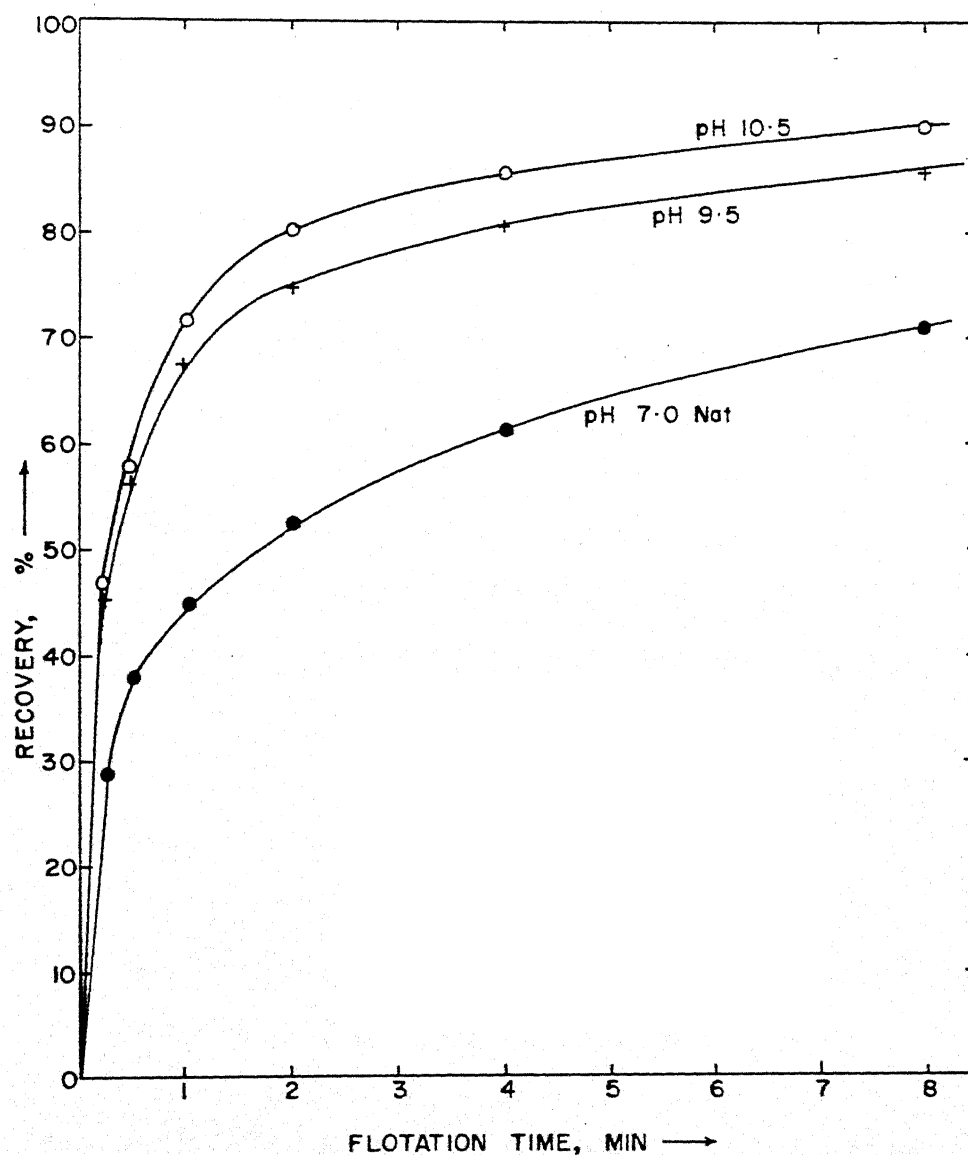


FIGURE 4.9 Effect of pH on Flotation Recovery (grinding time 5 min; xanthate 7 ml; MIBC 3 drops; rpm 700; air flow 5 lpm).

weight floated with pH, indicating an improvement in grade and hence in selectivity.

The flotation rate distribution estimated by the model analysis is shown in Figure 4.10 for different pH conditions. The rate distributions shifted towards right with the increase in pH, indicating an improvement in flotation rate as the pH was increased.

4.3.5 Effect of Xanthate Addition

In order to study the effect of collector dosage on the flotation behaviour of copper minerals, experiments were carried out with xanthate quantities of 4, 7, 9 and 12 ml of 1 percent solution. All the other conditions were maintained at the same level. The results of these experiments are presented in Table 4.8 and the recovery-flotation time plots are shown in Figure 4.11. The copper recovery consistently improved when the xanthate quantity was increased from 4 ml to 12 ml. In the case of weight recovery, a substantial increase was observed when xanthate was increased from 4 to 7 ml. Afterwards the increase did not result in any significant change in the weight floated. In order to find out if any further increase in xanthate quantity would bring about improvement in flotation behaviour, recovery values were plotted against xanthate quantity for different flotation times and are shown in Figure 4.12. It may be seen that at the end of 17 minutes (Rougher + Scavenger), the recovery has already stabilised. For other flotation times, the recovery is in the process of

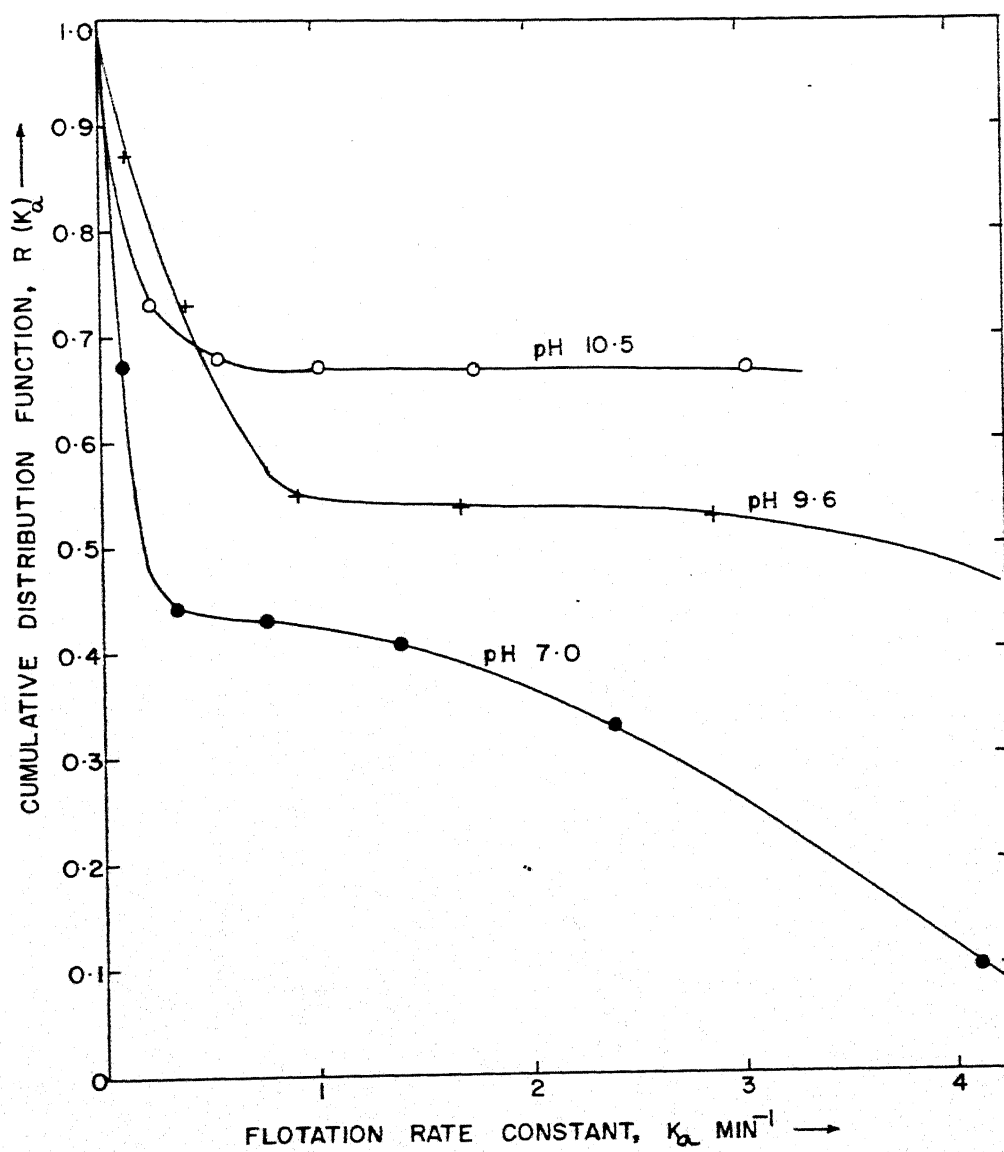


FIGURE 4.10 Effect of pH on Cumulative Apparent Flotation Rate Constant Distribution.

TABLE 4.8

Effect of Xanthate Quantity on Flotation Behaviour

Float Fraction	Cum. Time min	Cum. Unit Weight				Cum. Copper Recovery, %			
		Expt. 12	Expt. 4	Expt. 13	Expt. 14	Expt. 12	Expt. 4	Expt. 13	Expt. 14
F1	0.25	0.7	1.3	0.9	1.7	11.3	28.9	23.1	43.5
F2	0.5	1.0	1.8	1.6	2.4	17.0	37.8	38.8	57.3
F3	1.0	1.4	2.4	2.3	3.0	23.5	44.8	52.4	66.7
F4	2.0	1.9	3.3	3.6	3.5	31.8	52.1	67.9	73.1
F5	4.0	3.2	4.9	5.3	4.3	45.6	61.3	76.4	78.5
F6	8.0	5.2	7.7	7.2	6.3	60.3	70.9	82.1	86.2
Xanthate, ml	4	7	9	12	4	7	9	12	

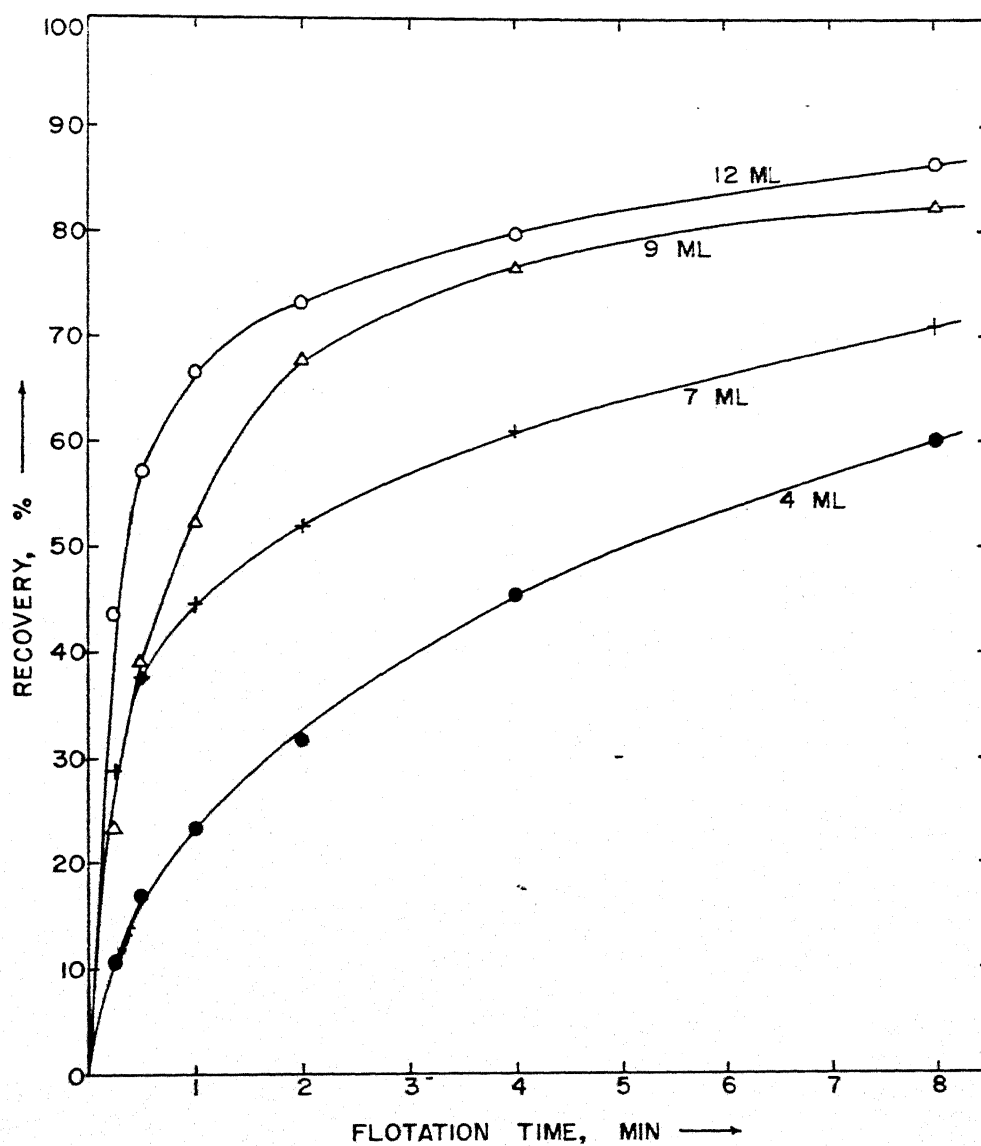


FIGURE 4.11 Effect of Xanthate Quantity on Flotation Recovery (grinding time 5 min; MIBC 3 drops; pH 7; rpm 700; air flow 5 lpm).

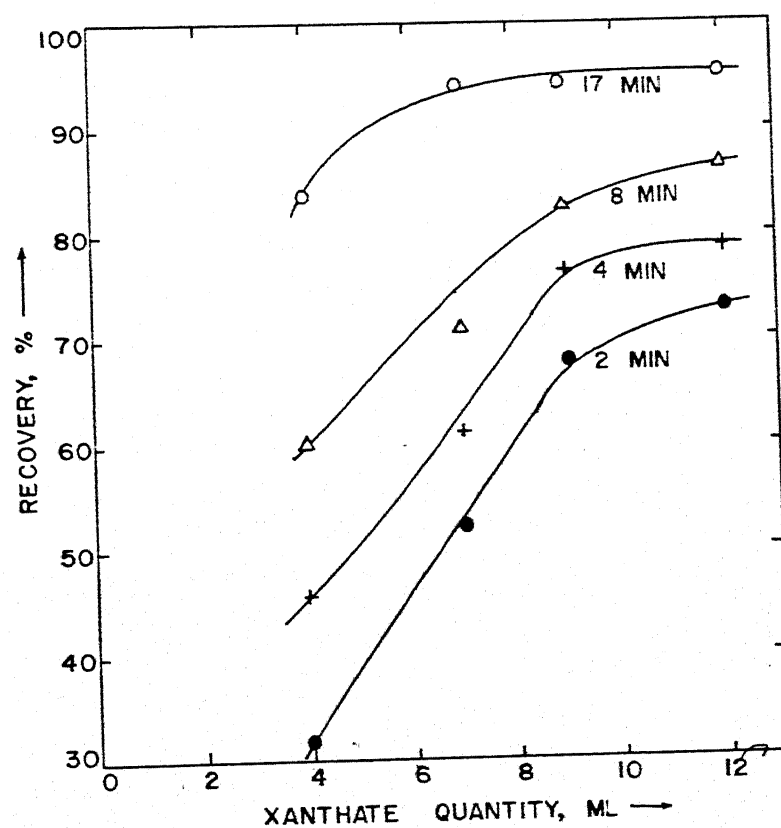


FIGURE 4.12 Effect of Xanthate Quantity (recovery-xanthate quantity plot).

getting stabilised and further addition of xanthate would not significantly produce any increase in recovery.

The flotation rate distributions are shown in Figure 4.13. With increase in collector quantity, the distributions of flotation rates were shifted significantly towards right, explaining the observed flotation behaviour. However, the distribution for 9 ml xanthate did not show this trend (shown in dotted lines). This could be only due to the common experimental error encountered in semi-batch flotation tests on account of the possible difference between the experimental and effective flotation times (Marris and Chakravarty, 1970). It may be seen from Table 4.8 that while there is a definite increasing trend of weights floated, with the increase in xanthate addition, it is different only in the case of the experiment with 9 ml xanthate (Expt. 13) and it has been already mentioned that $R(K_a)$ is highly sensitive to $M_p(t)$ values.

4.3.6 Effect of Frother Addition

All the batch experiments were normally carried out with the addition of frother being 3 drops of MIBC. In order to see if an increase in frother dosage would affect the flotation behaviour, an exploratory experiment was conducted with 5 drops of MIBC. The results are presented in Table 4.9. There was improvement in both weight of the float and the copper recovery. However with 5 drops of MIBC, initially the froth thickness was very small and scraping was difficult. This difficulty lessened after some time when the frother concentration sufficiently decreased by froth removal from the liquid-air

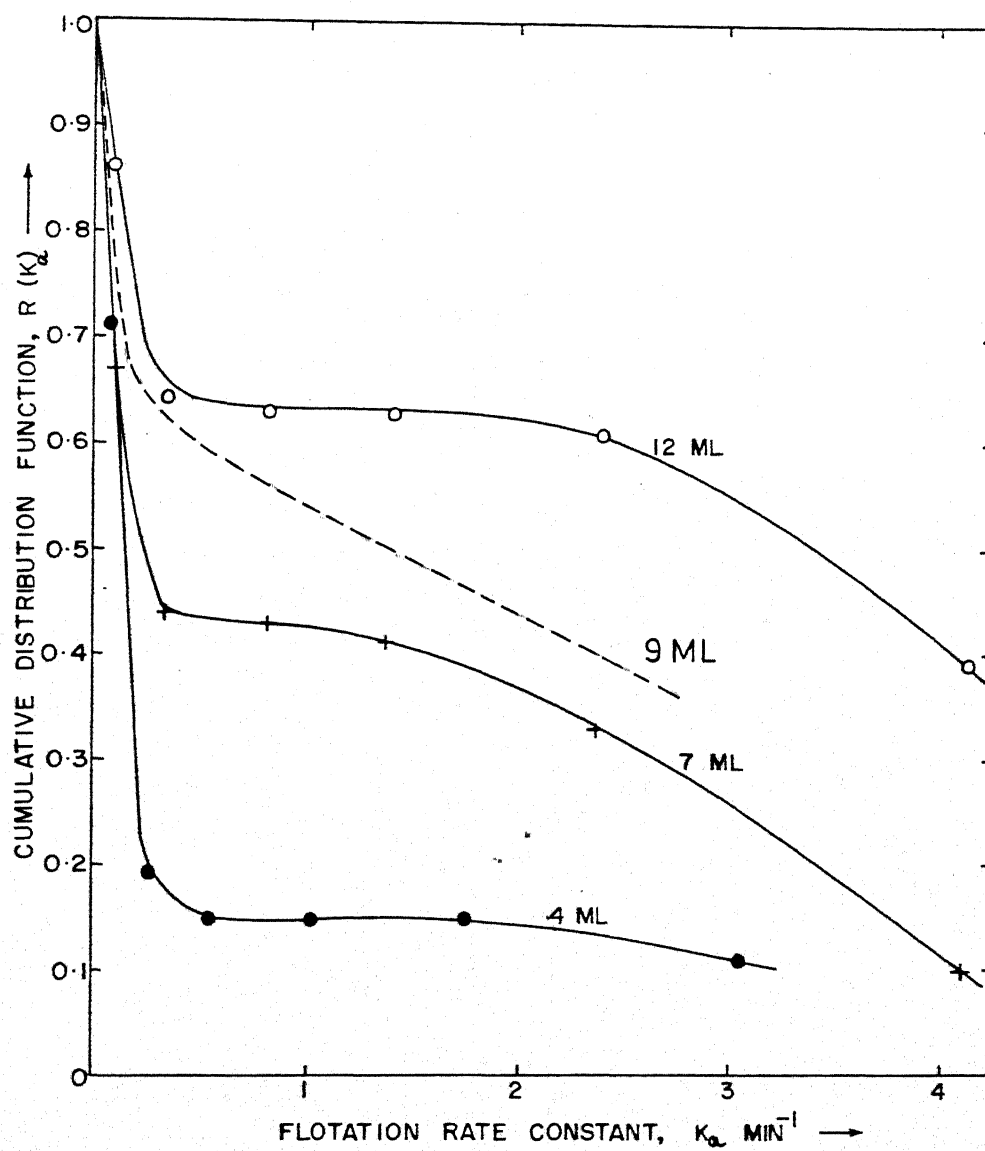


FIGURE 4.13 Effect of Xanthate Quantity on Cumulative Apparent Flotation Rate Constant Distribution.

TABLE 4.9

Effect of Frother Quantity on Flotation Behaviour

Float Fraction	Cum. Time min	Cum. Unit Weight		Cum. Copper Recovery, %	
		Expt. 4	Expt. 15	Expt. 4	Expt. 15
F1	0.25	1.3	1.8	28.9	45.4
F2	0.5	1.8	2.6	37.8	58.3
F3	1.0	2.4	3.4	44.8	67.4
F4	2.0	3.3	4.6	52.1	73.7
F5	4.0	4.9	6.3	61.3	79.2
F6	8.0	7.7	8.3	70.9	84.5
MIBC, drops		3	5	3	5

interface. The variation of flotation rate distribution due to increase in frother quantity is shown in Figure 4.14. The distribution shifts towards right with the increase in frother and it becomes less sharp indicating that the frother, also increases the proportion of slow floating species due to loss in selectivity.

4.3.7 Effect of Impeller Speed

The impeller speed affects the dispersion of particles and air bubbles in the flotation cell and increases the probability of bubble-particle collision. In sub-aeration type flotation cells, the shaft rpm bears a direct relation to the aeration rate also. Leeds cell provides an excellent control over this variable and the effect of rpm on flotation behaviour can be studied reliably and accurately. Experiments were carried out with three different impeller speeds, 700, 900 and 1000 rpms. The results of these experiments, given in Table 4.10, show that when the rpm was increased from 700 to 900, the copper recovery increased (Figure 4.15) but the change from 900 to 1000 rpm did not produce any significant increase in recovery. The recovery-rpm plots (Figure 4.16) also prove this observation and indicate that any further increase in rpm would not improve the copper recovery. Very high rpm may have detrimental effect as high turbulence will lead to breakage of bubble-particle assemblies resulting in lower recoveries.

The flotation rate distributions for these experiments are shown in Figure 4.17. The rates improved initially when

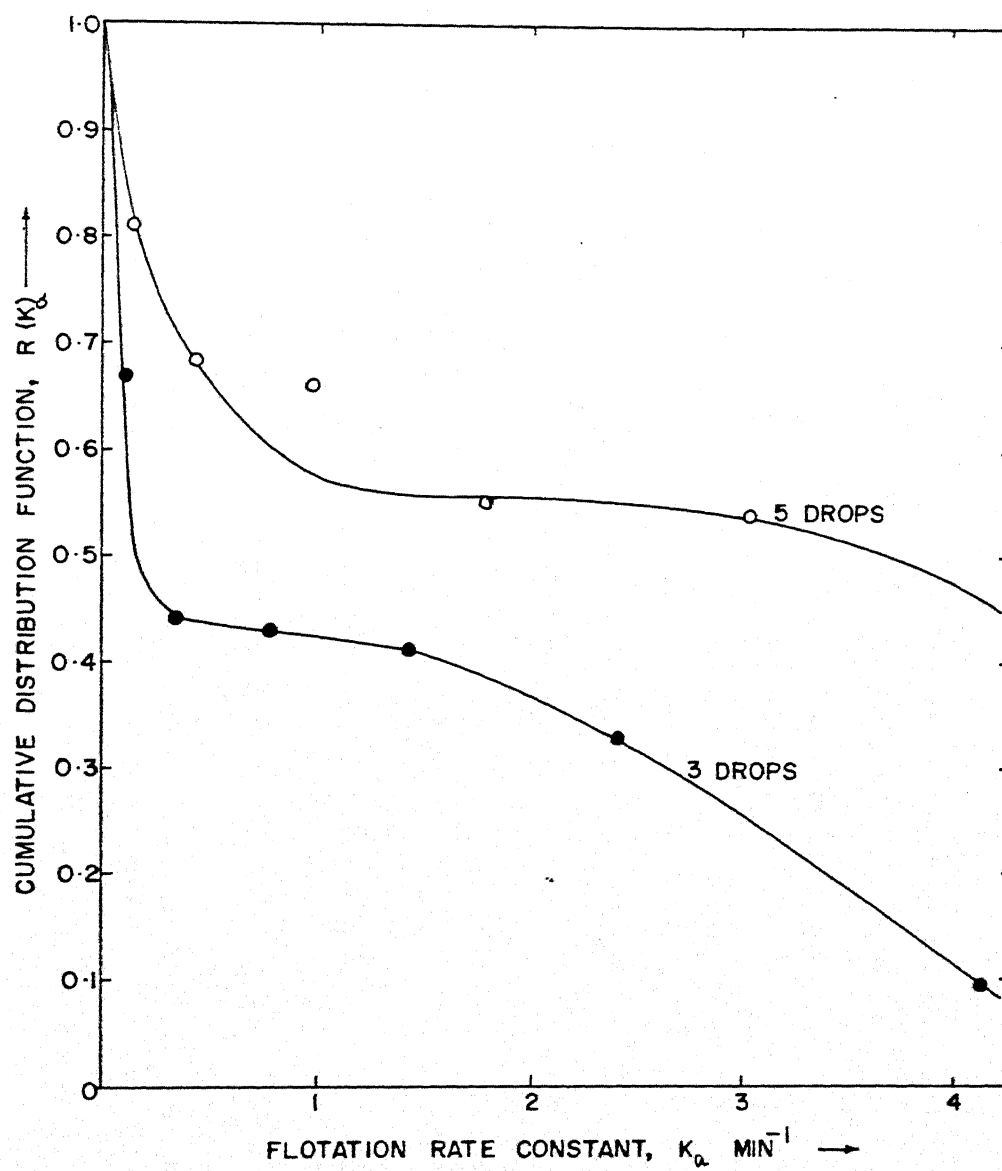


FIGURE 4.14 Effect of Frother Quantity on Cumulative Apparent Flotation Rate Constant Distribution.

TABLE 4.10
Effect of RPM on Flotation Behaviour

Float Fraction	Cum. Time min	Cum. Unit Weight				Cum. Copper Recovery, %			
		Expt. 4	Expt. 17	Expt. 27	Expt. 27	Expt. 4	Expt. 17	Expt. 27	Expt. 27
F1	0.25	1.3	2.6	1.9	28.9	54.5	44.4		
F2	0.5	1.8	3.3	2.8	37.8	63.9	61.3		
F3	1.0	2.4	3.9	3.2	44.8	68.0	66.2		
F4	2.0	3.3	4.7	4.0	52.1	72.8	71.9		
F5	4.0	4.9	5.9	5.5	61.3	77.5	78.2		
F6	8.0	7.7	7.7	6.9	70.9	82.4	83.4		
RPM		700	900	1000	700	900	1000		

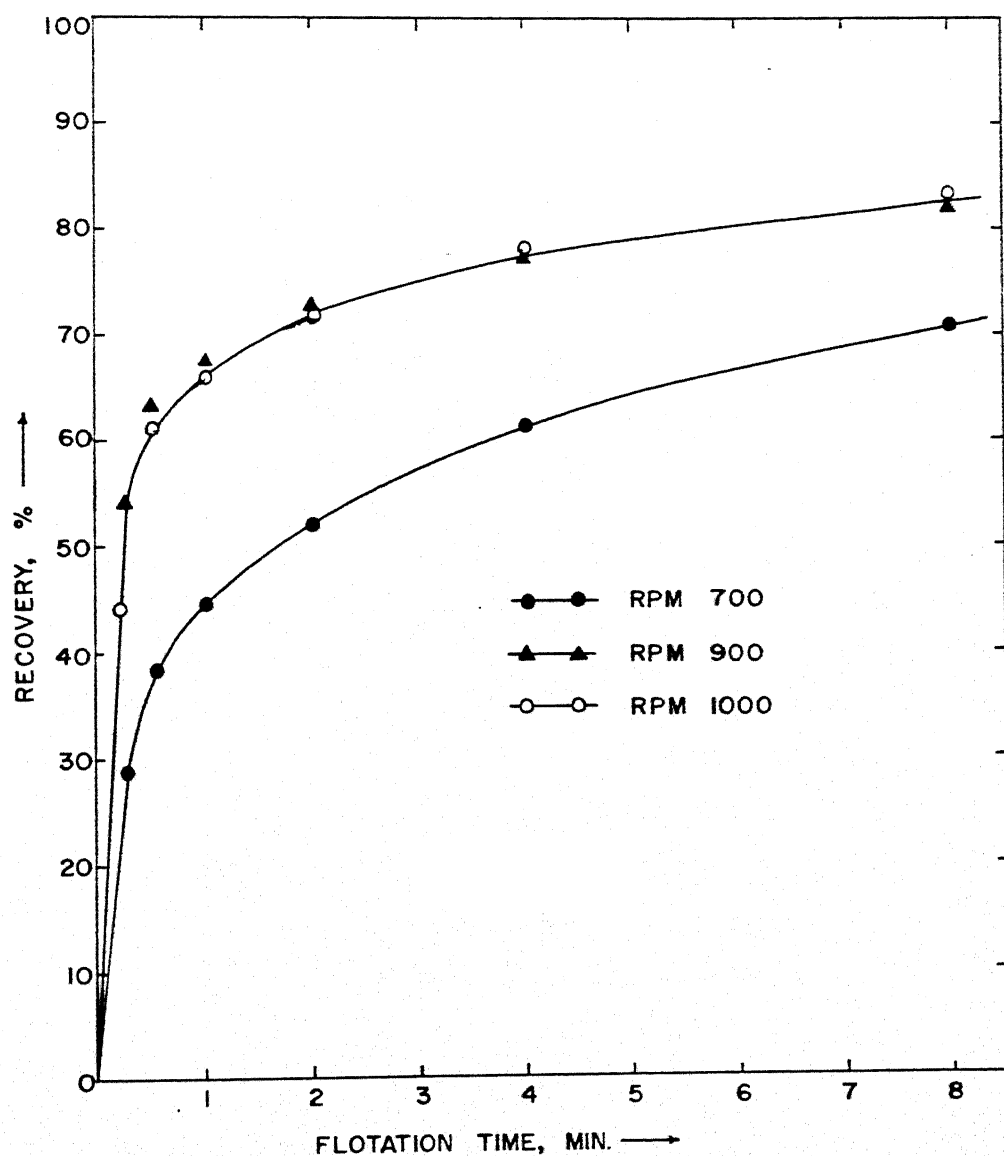


FIGURE 4.15 Effect of rpm on Flotation Recovery (grinding time 5 min; xanthate 7 ml; MIBC 3 drops; pH 7; air flow 5 lpm).

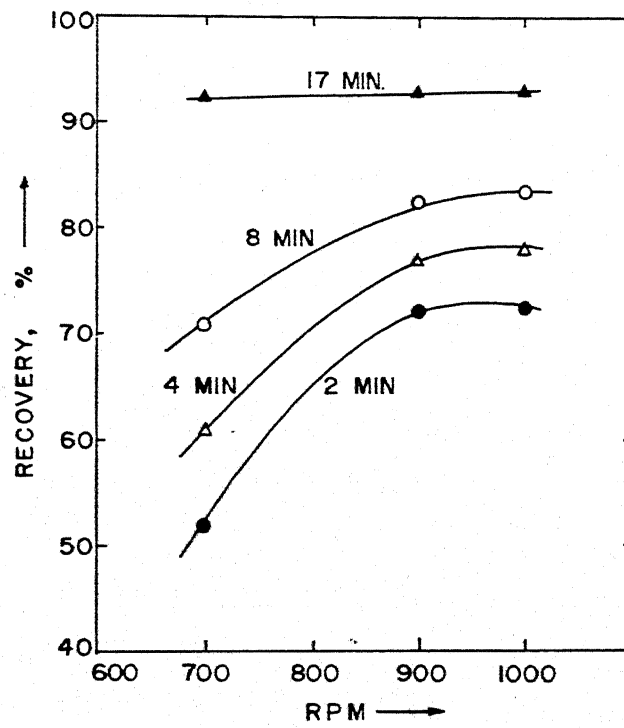


FIGURE 4.16 Effect of rpm (recovery-rpm plot).

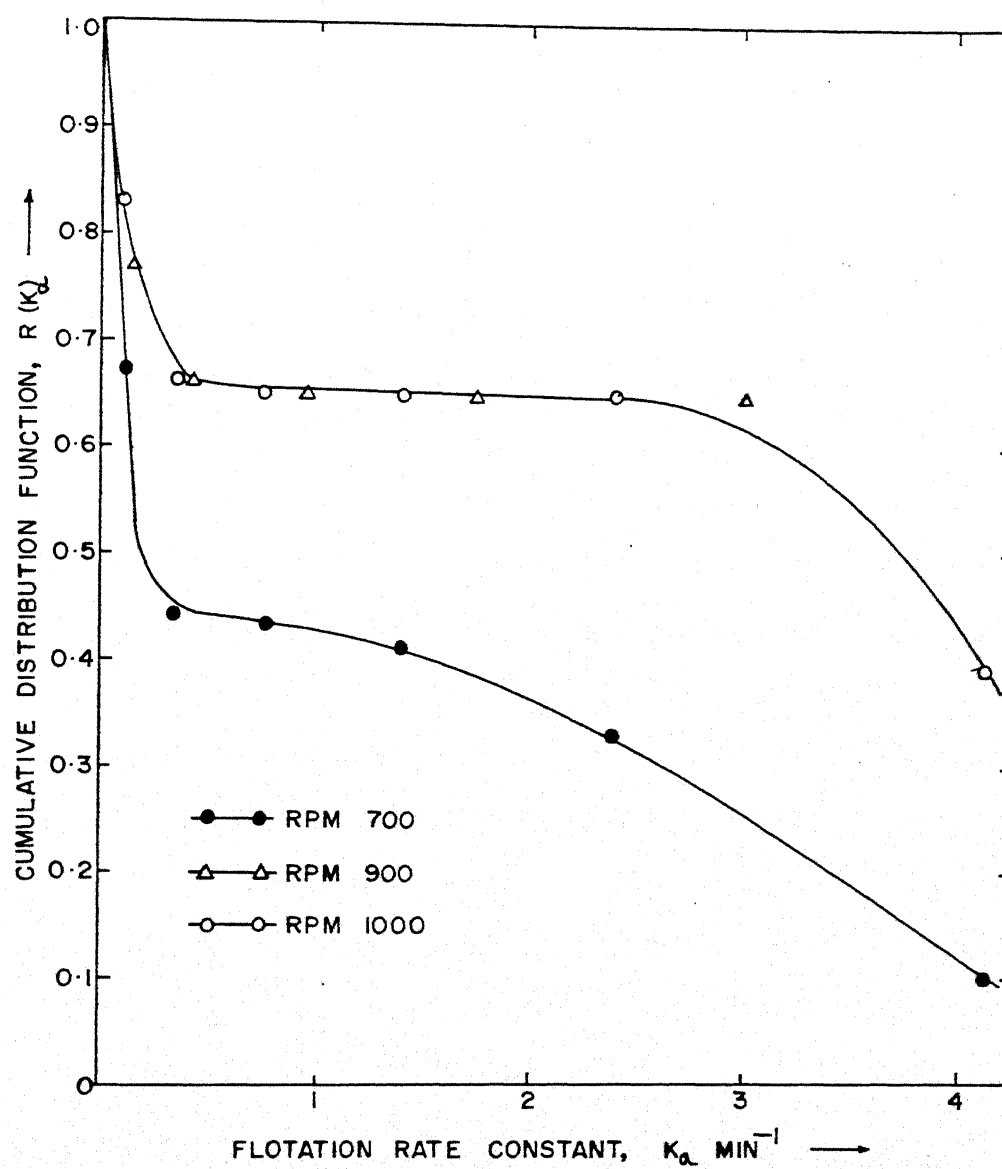


FIGURE 4.17 Effect of rpm on Cumulative Apparent Flotation Rate Constant Distribution.

the rpm was changed from 700 to 900 rpm and in the second step-increase in rpm, there was no change in flotation rates.

4.3.8 Effect of Aeration Rate

Aeration rate is an important equipment variable, which has been receiving much attention recently, especially in simulation using multiphase kinetic models. Froth characteristics depend very much on aeration rate. Increase in aeration rate increases the flotation rate (Mehrotra and Kapur, 1974). However, Laplante et al (1983) reported that as air flow rate was increased, initially the flotation rates increased, but later, decreased after passing through a maximum.

Experiments were conducted to study the effect of aeration rate on the flotation behaviour. Three different air flow rates, 5, 7 and 10 lpm were tried and the results are presented in Table 4.11. There was a consistent increase in weight floated and copper recovery with aeration rate (Figure 4.16). The flotation rates were improved throughout the range of aeration rate studied, in line with the observations of Kapur and Mehrotra (1974) (Figure 4.19).

4.3.9 Flotation Rate Distributions of Rougher, Scavenger and Cleaner Feeds

So far it has been the common practice to assume that the floating characteristics of particulate species during various stages of flotation namely, roughing, scavenging and cleaning, remain the same. However, because of varying nature of feed materials, varying environmental conditions in the

TABLE 4.11

Effect of Aeration Rate on Flotation Behaviour

Flotation Fraction	Cum. Time min	Cum. Weight Recovery		Cum. Copper Recovery, %		
		Expt. 4	Expt. 16	Expt. 21	Expt. 4	Expt. 16
F1	0.25	1.3	1.6	2.0	23.9	42.0
F2	0.5	1.8	2.1	2.6	37.8	52.6
F3	1.0	2.4	2.9	3.2	44.8	63.8
F4	2.0	3.3	3.7	4.1	52.1	70.0
F5	4.0	4.9	5.0	5.1	61.3	76.6
F6	8.0	7.7	6.9	7.3	70.9	83.2
Air Flow Rate, lpm	5	7	10	5	7	10

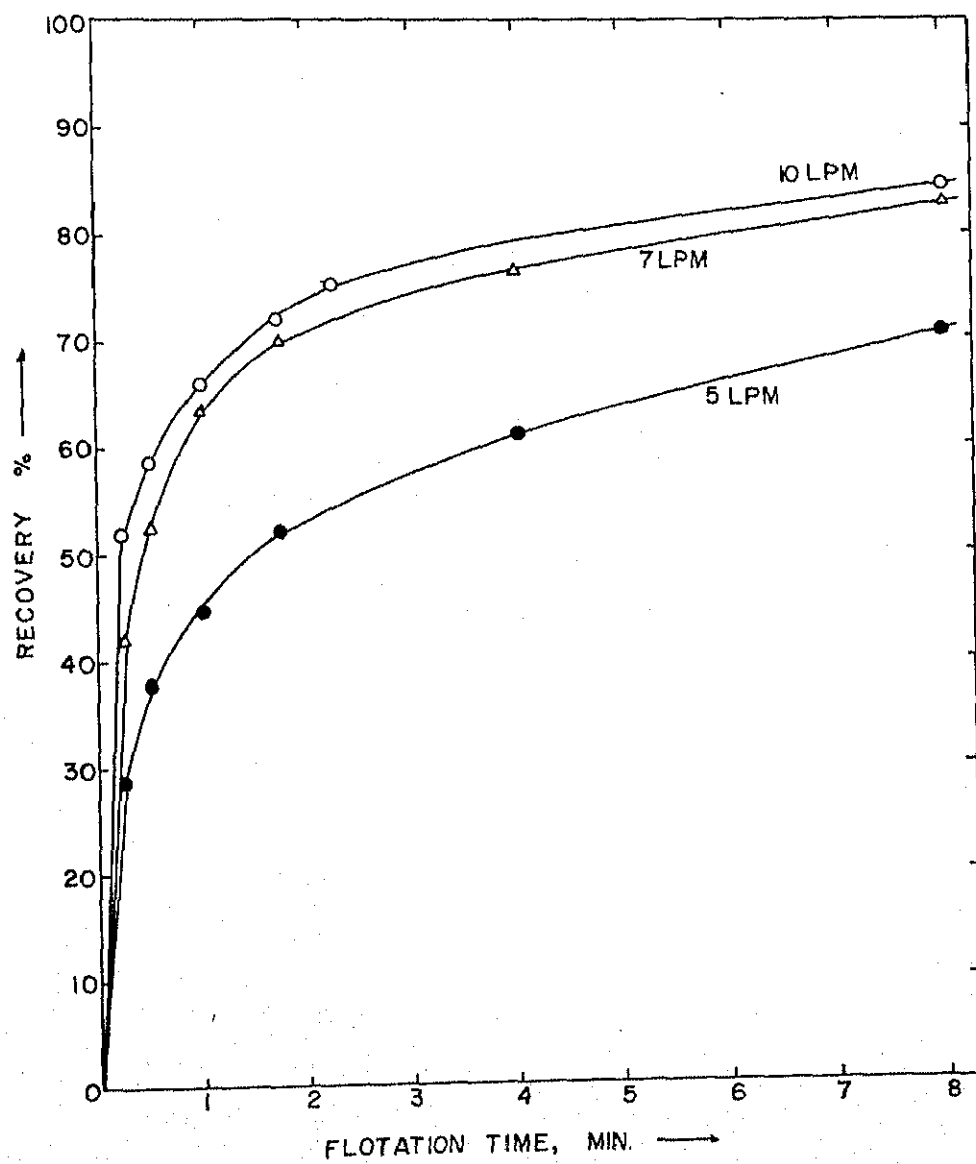


FIGURE 4.18 Effect of Aeration Rate on Flotation Recovery (grinding time 5 min; xanthate 7 ml; MIBC 3 drops; pH 7; rpm 700).

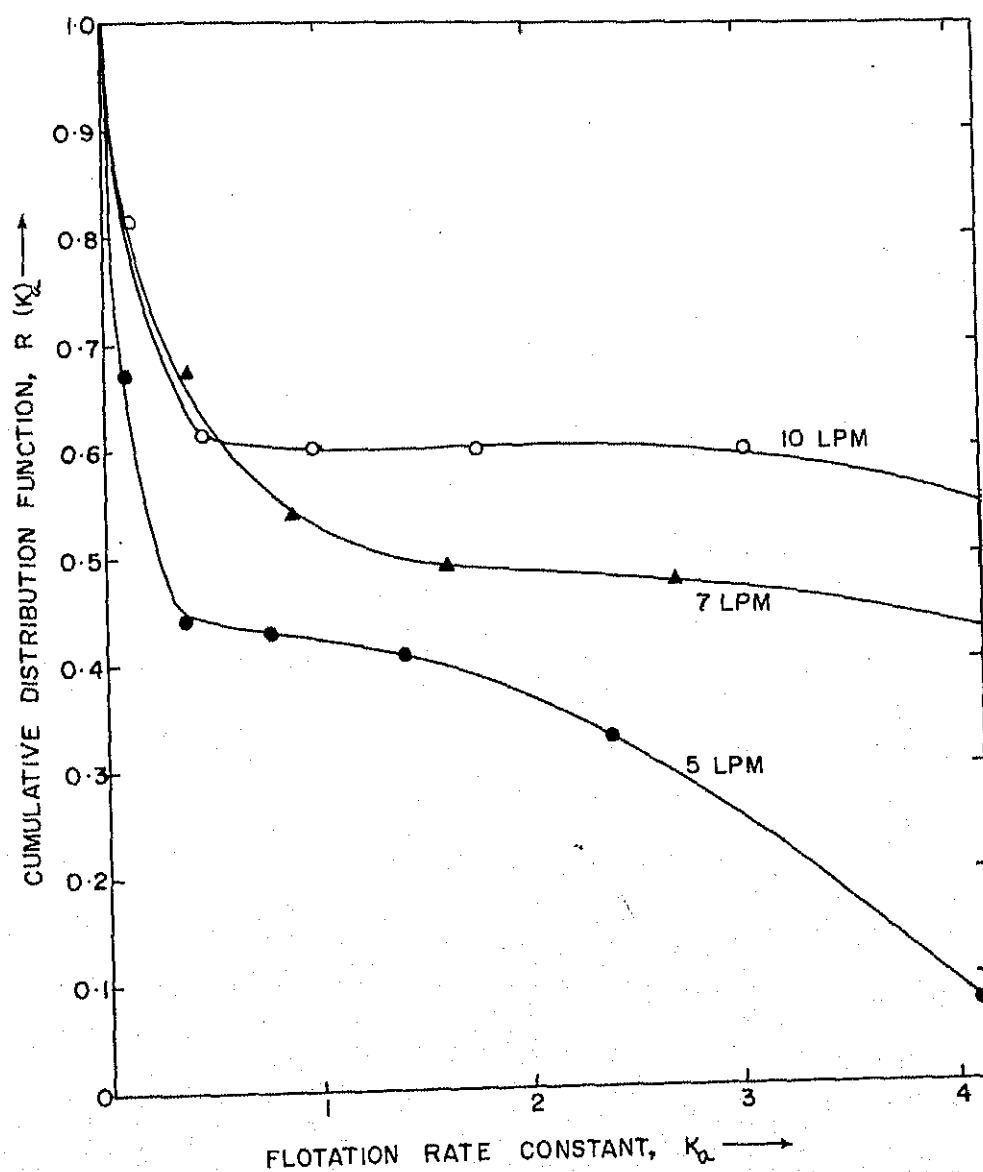


FIGURE 4.19 Effect of Aeration Rate on Cumulative Apparent Flotation Rate Constant Distribution.

different stages and varying operating conditions, there is reason to believe that the floating characteristics of particulate species in these various stages will not be the same. To establish this fact, batch experiments simulating two roughing, one scavenging and one cleaning stages were carried out.

The roughing and scavenging experiments were carried out in the usual manner, with the experimental conditions as mentioned in Table 3.2 for Expt. 25. But changes were made in the mode of float collection, according to which the collection was done in three time batches as follows:

0 - 4 min	Rougher 1
4 - 8 min	Rougher 2
8 - 17 min	Scavenger

In each time-batch, the float was collected on incremental time basis to generate kinetic data. As in other cases after 3 minutes of flotation time (i.e., after Rougher 2), reagent conditioning was done (xanthate - 7 ml of 1 percent solution, 3 drops of MIBC and conditioning for 4 minutes with xanthate and for one more minute after the addition of MIBC), and subsequently, the flotation was continued. The material balance was computed on the overall basis (Table 4.12) as well as on the basis of different stages. The cleaner flotation was carried out by refloating the floats obtained during the roughing stages. In order to maintain the same pulp density in cleaner slag also, as in roughing stages, sufficient amount of rougher float (or feed to cleaner stage) was collected from a number of batches of rougher flotation. About 5 batches were found to

TABLE 4.12
Overall Material Balance for Expt. 25

Float Fraction	Cum. Time min	Unit Weight	Copper Assay, Percent	Copper Recovery %
F1	0.25	1.6	22.33	35.4
F2	0.5	0.8	14.77	12.5
F3	1.0	1.0	8.74	8.3
F4	2.0	1.2	6.50	7.6
F5	4.0	1.6	4.43	7.0
F6	4.25	0.4	3.90	1.6
F7	4.5	0.3	3.72	1.2
F8	5.0	0.5	3.54	1.9
F9	6.0	0.6	3.54	2.2
F10	8.0	1.1	2.95	3.3
F11	8.25	0.5	6.85	3.6
F12	8.5	0.3	4.92	1.6
F13	9.0	0.5	3.35	1.8
F14	10.0	0.8	2.72	2.2
F15	12.0	0.9	1.97	1.8
F16	17.0	0.9	1.61	1.4
T		39.7	0.168	6.6

be adequate to get sufficient feed for cleaner flotation. The results of this experiment are given in Table 4.13.

The flotation rate distributions for the feeds to all the four stages (two roughers, one scavenger, and one cleaner) were independently estimated and are shown in Figure 4.20. It may be seen that the rate distribution curve for Rougher 1 shows the flotation rate constant to be distributed over a wide range, while that for Rougher 2 shows a narrow distribution. It is because most of the fast-floating species (species with higher rate constants) have been removed in the float. In the case of scavenger feed, the flotation rate distribution should have been shifted more towards left from Rougher 2 curve, indicating the presence of only very slow floating species (species with very low rate constants) in the cell. However, such a distribution was not obtained for scavenging stage simply because at the end of Rougher 2 stage, fresh reagent conditioning was done and this caused the distribution to shift towards right, and to appear in the position as shown in Figure 4.20.

The rate constant distribution of the feed to the cleaner stage is also shown in the same figure. Contrary to the expectation, it was found that the cumulative distribution curve for cleaner flotation - wherein about 70 percent of the total weight has floated - was lower than that for Rougher 1. It is likely due to the fact that the percent copper recovery values D_i were used as the input for the computer program to evaluate the rate distribution, and since the recovery values are computed with respect to the total amount of copper present

TABLE 4.13

Results of Cleaner Flotation (Expt. 26)

Float Fraction	Cum. Time min	Weight %	Copper Assay %	Copper Recovery %
F1	0.25	11.5	25.91	32.5
F2	0.5	4.6	25.78	12.9
F3	1.0	4.2	24.51	11.2
F4	2.0	8.7	16.64	15.8
F5	4.0	9.4	9.91	10.2
F6	8.0	11.8	5.27	6.8
F7	15.0	15.9	3.37	5.8
F8	17.0	2.4	2.35	0.6
T		31.5	1.19	4.2

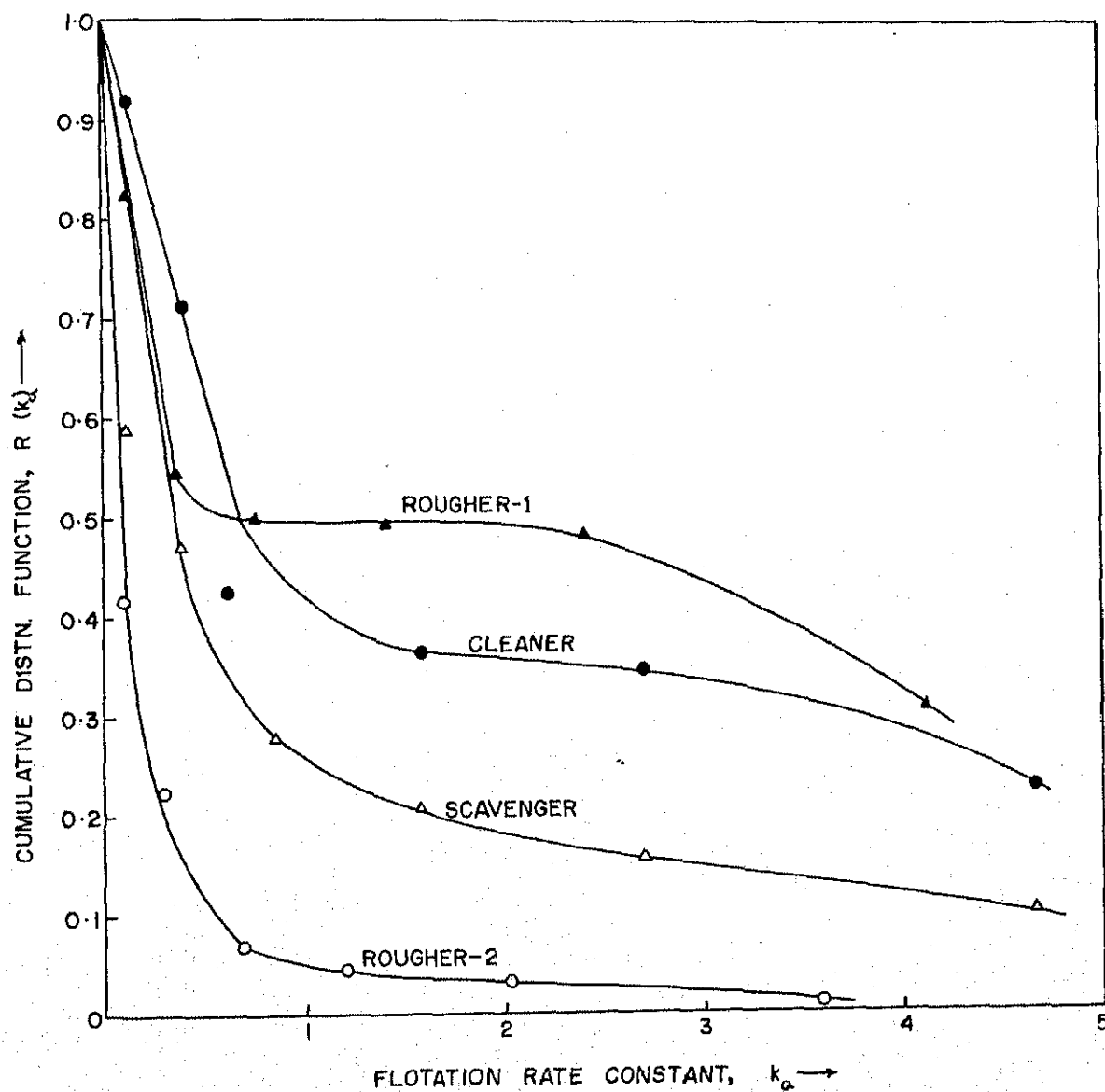


FIGURE 4.20 Cumulative Rate Constant Distribution of Feed to Rougher, Scavenger and Cleaner Stages (on total copper basis).

in the feed to that stage, there is not much difference in the values obtained for rougher and cleaner flotations. It should, however, be recognised that the major difference between the two stages lies in the concentrate weight, which is much higher in the case of cleaner. To resolve this anomaly between the rate distributions for rougher and cleaner stages, the data was reanalysed using: (i) the total weight of the feed as the basis and, (ii) the total weight of floatable material as the basis. The results obtained for these two cases are presented below.

Rate Constant Distribution on Total Weight Basis: It was attempted to estimate the rate distribution using the concentrate weight percent as the input data, instead of percent copper recovery. This was done for a single set of data obtained for experiments wherein grinding time was varied (cf Section 4.3.2) and, the results are shown in Figure 4.21. It can be seen that the trend, which was obtained in Figure 4.2 is followed here as well but the curves are shifted to the bottom, due to the fact that the percent weight of material floated is much less in comparison to the percent copper recovered in any time interval.

Rate Constant Distribution on Total Floatable Material Basis: Since the feed in all the experiments consisted of very large amounts of non-floatable material, it was decided to take into account the fraction of non-floatable material in the feed separately. The modified numerical method, proposed by Kapur and Mehrotra (1974) was used for this purpose. The

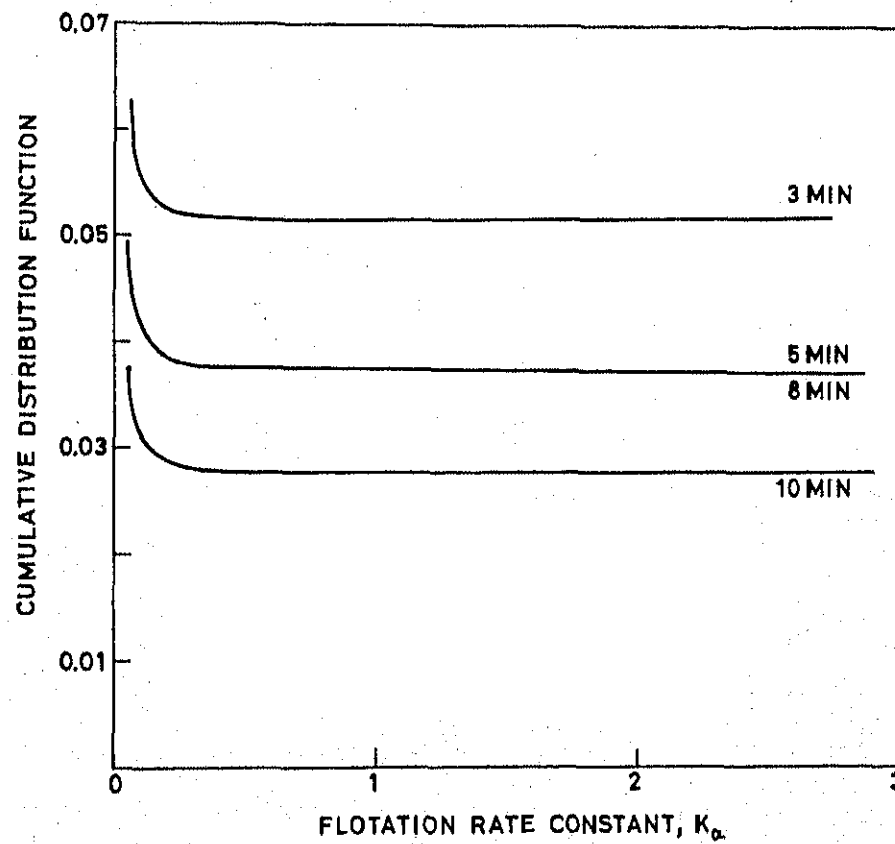


FIGURE 4.21 Effect of Grinding Time on Rate Constant Distribution (on total weight basis).

important feature of this method is that it directly gives the amount of non-floatables in the feed. The results obtained for a few experimental runs, in which grinding time was varied, are shown in Figure 4.22. In this case also it is seen that the feed material ground for 3 minutes gave the highest rate and, the one with 10 minutes grinding, the lowest.

By extrapolation of the rate distribution curve to the y axis (rate constant = 0), the amount of non-floatable material in the feed can be obtained. It can be seen from the figure that the material ground for 3 minutes had larger amounts of fast floating species, but had only about 20 percent of the total feed material as floatable. As grinding time was increased, the rate distribution curve shifts towards left indicating the decrease of the amount of fast floating species in the feed while the amount of total floatable species increased. At 10 minutes grinding the floatable species rose to about 42 percent from 20 percent for 3 minutes grinding.

Analysis of Rougher-Scavenger-Cleaner Experiments on Total Floatable Material Basis: The results of the rougher-scavenger-cleaner experiments were reanalysed using the technique described above and the rate distributions were estimated (Figure 4.23). The earlier anomaly of the cleaner curve being lower than Rougher 1 curve, has been removed in this case. The cleaner curve is much above the rougher curve and also showed that the cleaner feed contained only about 15 percent by weight as non-floatable. The Rougher 1 curve is similar to cleaner curve in shape but has much higher amount of non-floatables (almost 75 percent). In the case of Rougher 2, the

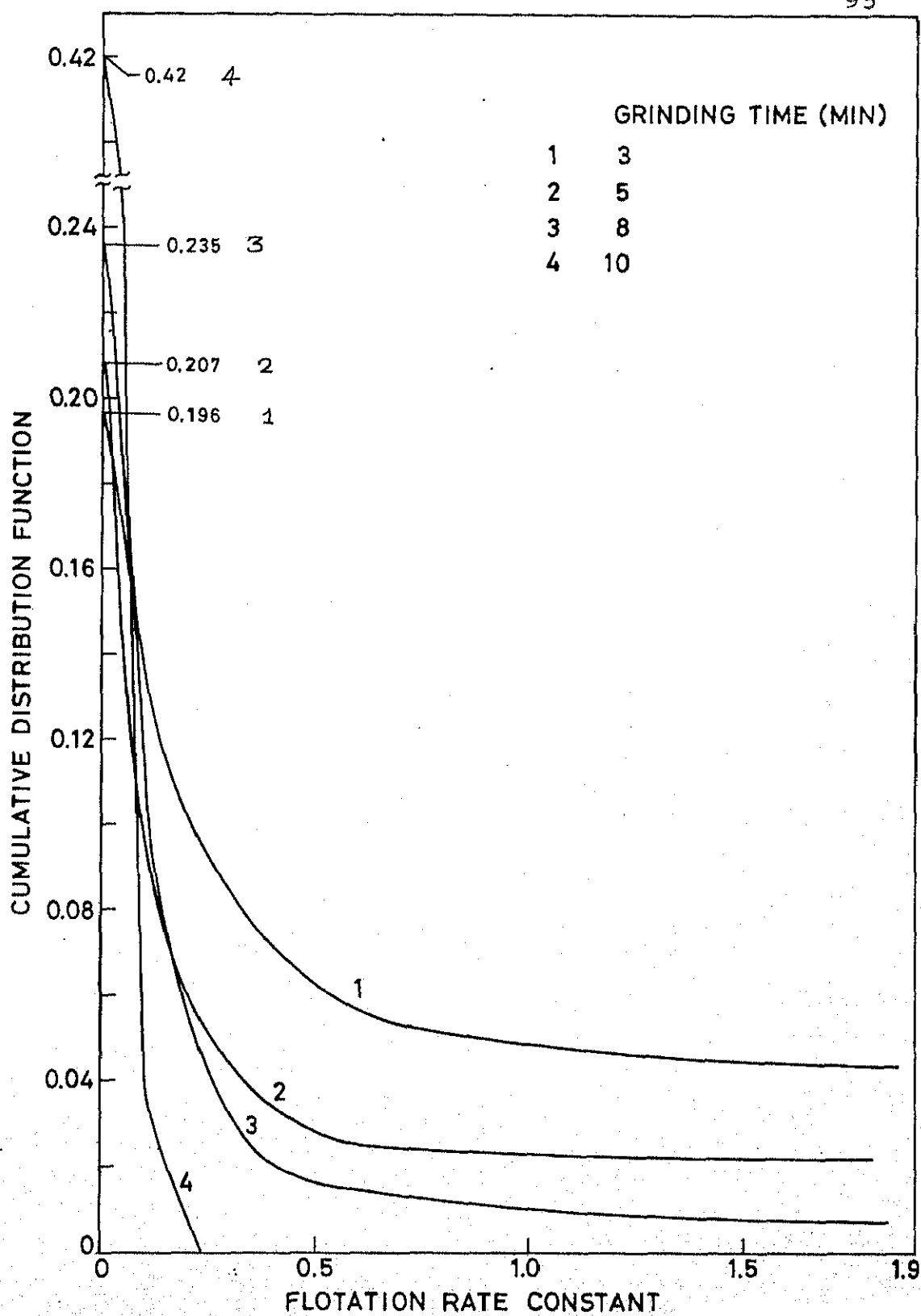


FIGURE 4.22 Effect of Grinding Time on Rate Constant Distribution (on total floatable weight basis).

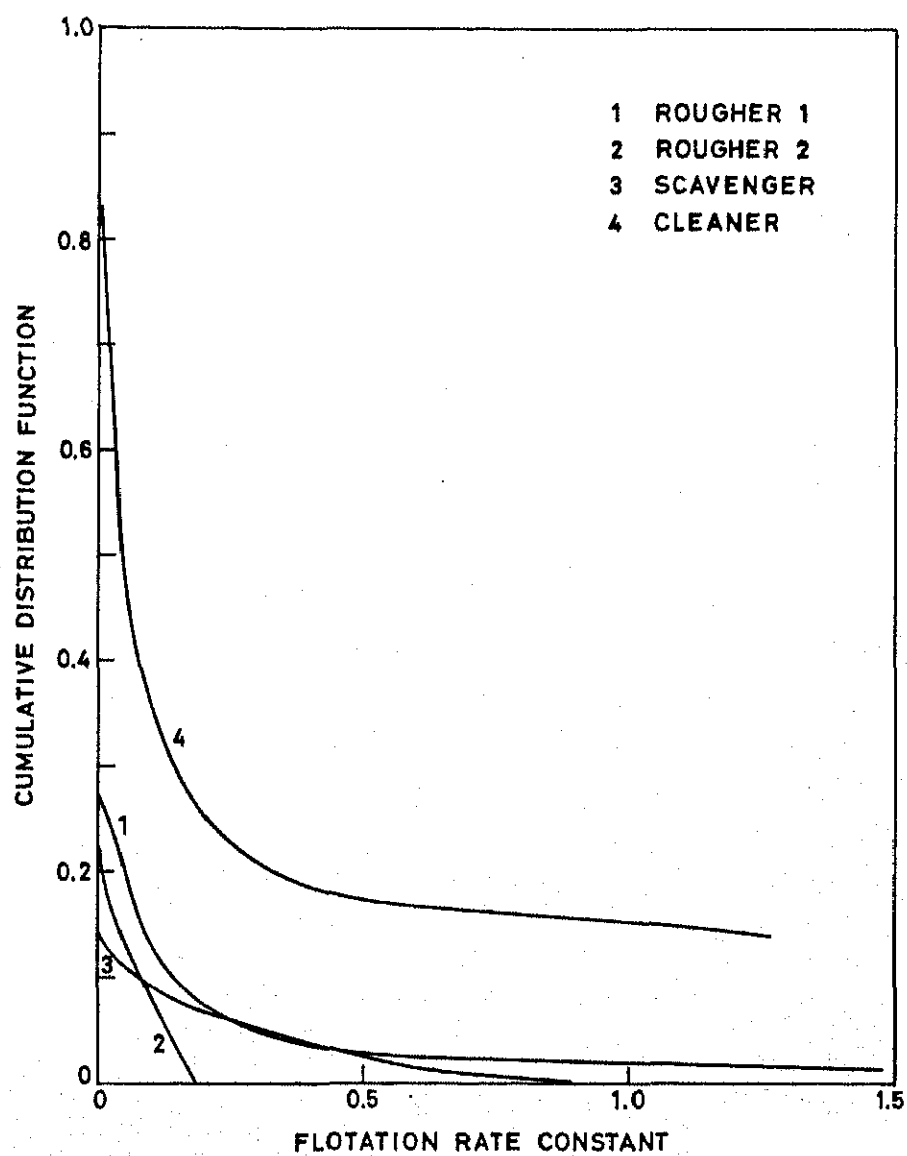


FIGURE 4.23 Cumulative Rate Constant Distribution of Feed to Rougher, Scavenger and Cleaner Stages (on total floatable weight basis).

curve is shifted to the left and has still higher percent of non-floatables (80 percent) as most of the floatable materials have been removed in the earlier stage. In the case of scavenger curve, the amount of non-floatables has increased still further (85 percent), but the curve is shifted upwards because of reagent conditioning.

4.4 ALTERNATE METHOD OF DETERMINING RATE CONSTANT DISTRIBUTION

In the numerical method proposed by Rapur and Mehrotra (1974) to estimate the rate constant distribution, the rate constant K_a is assumed to have a range between 0 and ∞ . However, physically it is more meaningful if the maximum value of K_a is some finite value (K_{am}) and not infinity. In what follows is an alternate method of determining rate constant distribution in which K_a is assumed to vary between 0 and K_{am} .

For K_a varying between 0 and K_{am} Eq. 4.9 is modified in the following manner.

$$\bar{M}_p(t) = \int_0^{K_{am}} R(K_a) \exp(-K_a t) dK_a \quad (4.11)$$

To change the limit of the integral to -1 to +1 (as done by Rapur and Mehrotra) following transformation may be carried out. Let

$$x = \frac{2K_a}{K_{am}} - 1 \quad (4.12)$$

This satisfies the requirement, that when $K_a = 0$, x should be -1 and when $K_a = K_{am}$, x should be +1. Substitution of Eq. 4.12 in Eq. 4.11 gives

$$\bar{M}_p(t) = \frac{K_{am}}{2} \int_{-1}^{+1} R \left[\frac{K_{am}(1+x)}{2} \right] \exp \left[- \frac{K_{am}(1+x)}{2} t \right] dx \quad (4.13)$$

Discretization using n-point Lobatto quadrature gives

$$\bar{M}_p(t) = \frac{K_{am}}{2} \left[2 \frac{F(1) + F(-1)}{n(n-1)} + \sum_{i=2}^{n-1} H_i F(x_i) \right] \quad (4.14)$$

$$\text{where } F(x_i) = L(x_i) \exp \left[- \frac{K_{am}(1+x_i)}{2} t \right] \quad (4.15)$$

$$\text{and } L(x_i) = R \left[\frac{K_{am}(1+x_i)}{2} \right] \quad (4.16)$$

It can be shown that

$$F(1) = 0 \quad \text{and} \quad F(-1) = 1 \quad (4.17)$$

Now Eq. 4.14 becomes

$$\bar{M}(t) = \frac{K_{am}}{n(n-1)} + \frac{K_{am}}{2} \sum_{i=2}^{n-1} H_i F(x_i) \quad (4.18)$$

This equation can be solved for $L(x_i)$ by any optimisation technique, using the least square objective function of the type

$$E = \sum_{j=1}^N \left[\bar{M}(t) - \frac{K_{am}}{n(n-1)} - \frac{K_{am}}{2} \sum_{i=2}^{n-1} H_i F(x_i) \right]^2 \quad (4.19)$$

All the constraints that were applicable in the earlier treatment (Appendix A1) are applicable here too. A search for K_{am} can be made to get the best fit.

This transformation has been incorporated in a suitable program and estimation of $R(K_a)$ has been carried out for a number of experiments. With this transformation also, a close

fit, comparable to the earlier method is obtained. The advantages of this transformation are that (1) it gives a more physically meaningful upper limit for apparent flotation rate constant, which can be obtained by a coarse, followed by a fine search and (2) the scaling factor, s need not be used.

CHAPTER 5

CONCLUSIONS

Semi-batch flotation experiments carried out on Malanjkhand copper ore, using Leeds open-top flotation cell indicate that good reproducibility of test results is obtained with this cell.

Analysis of experimental results with the help of distributed apparent flotation rate constant-kinetic model show that it is not necessary to assume standard statistical distribution for the rate constants. It is confirmed that actual distribution can be obtained in the cumulative form by the numerical inversion of Laplace transform using Lobatto quadrature formula and that this model gives a very close fit with the experimental data.

As grinding time was increased, the copper recovery was found to decrease much against the common notion that as grinding time was increased, degree of liberation increased which caused increase in recovery. On the other hand increased grinding caused the increased production of -200 mesh material from which the copper recovery is sub-optimal.

Particle size affects the flotation behaviour to a great extent. Out of three size ranges studied, -100+200 mesh size fraction gave the maximum copper recovery, which decreased both in coarser (-35+100 mesh) and in finer fraction (-200 mesh).

The flotation performance certainly improved when pH was increased. Higher recovery with better selectivity was obtained with increase in pH.

When the collector quantity was increased from 7 to 12 ml the recovery was found to increase and the results showed that further increase in collector quantity would not improve the flotation recovery. Indicative experiment conducted with increased quantity of frother indicated that copper recovery improved though with the loss of selectivity.

Studies conducted with the increasing impeller speed indicate that the flotation performance improved when the rpm was increased from 700 to 900 and remained constant on further increase.

When the aeration rate was increased from 5 to 10 lpm, the flotation behaviour improved throughout the range studied.

Experiments conducted simulating the rougher-scavenger-cleaner stages showed how the flotation rate distribution varied in all these stages. Estimation of these rate distribution for these stages gave more meaningful results when the numerical inversion was carried out on total floatable material weight basis using Legendre quadrature formula, taking into account the presence of non-floatable material in the feed.

An alternate numerical method for determining rate constant distribution is proposed. In this method it is assumed that the rate constant k_a has a range between 0 and k_{am} only and not 0 and ∞ as assumed by Kapur and Mehrotra.

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APPENDIX A1

In the method proposed by Kapur and Mehrotra (1974) the numerical inversion of the Laplace transform is carried out in the cumulative distribution mode. If $R(K_a)$ is the cumulative distribution function

$$M_p(K_a) = - \frac{dR(K_a)}{dK_a} \quad (A.1)$$

Substitution of Eq. A.1 in Eq. 4.7, followed by integration by parts, gives

$$\bar{M}_p(t) = \int_0^{\infty} R(K_a) \exp(-K_a t) dK_a \quad (A.2)$$

where

$$\bar{M}_p(t) = \frac{1 - H(t)}{t} \quad (A.3)$$

Eq. A.2 is the cumulative distribution analogue of Eq. 4.7. By definition the Laplace transform is

$$\bar{M}_p(t/s) = \int_0^{\infty} R(K_a) \exp(-K_a (t/s)) dK_a \quad (A.4)$$

where s is an adjustable scaling factor, ($s > 0$). The evaluation of integral was attempted by 3-point Lobatto quadrature, which has limits -1 and 1. In order to use Lobatto quadrature, the integral in Eq. A.4 has to be transformed suitably so that it will have limits -1 and 1. So let

$$x = 2 \exp\left(-\frac{K_a}{s}\right) - 1 \quad (A.5)$$

Substitution of Eq. A.5 in Eq. A.4 gives

$$\bar{M}_p(t/s) = \frac{s}{2^t} \int_{-1}^1 R(s \ln \frac{2}{x+1}) (x+1)^{t-1} dx \quad (A.6)$$

Discretisation of the integral in Eq. A.6 by n-point Lobatto quadrature gives

$$\bar{M}_p(t/s) = \frac{s}{2^t} \left[2 \frac{F(1) + F(-1)}{n(n-1)} + \sum_{i=2}^{n-1} H_i F(x_i) \right] \quad (A.7)$$

where H_i and x_i are, respectively the weights and abscissas of the Lobatto quadrature, given in Table A1, and

$$F(x_i) = L(x_i)(x_i + 1)^{t-1} \quad (A.8)$$

$$\text{where } L(x_i) = R(s \ln \frac{2}{x_i + 1}) \quad (A.9)$$

It can be easily shown that

$$F(1) = 2^{t-1} \quad \text{and} \quad F(-1) = 0 \quad (A.10)$$

Substitution of Eq. A.10 in Eq. A.7 gives

$$\frac{\bar{M}_p(t/s)}{s} = \frac{1}{n(n-1)} + \frac{1}{2^t} \sum_{i=2}^{n-1} H_i L(x_i)(x_i + 1)^{t-1} \quad (A.11)$$

Eq. A.11 can be solved for $L(x_i)$ and hence for $R(K_{ai})$ since x_i and K_{ai} are related by Eq. A.5, using some optimisation technique. A least square objective function of the following form was used

$$E = \sum_{j=1}^N \left[\frac{\bar{M}_p(t/s)}{s} - \frac{1}{n(n-1)} - \frac{1}{2^t} \sum_{i=2}^{n-1} H_i L(x_i)(x_i + 1)^{t-1} \right]^2 \quad (A.12)$$

where N is the total number of experimental data points and $j = 1, 2, \dots, N$.

The constraints are

$$(i) \quad R(K_{a1}) \geq 0, \quad 0 \leq K_{a1} < \infty \quad (A.13)$$

(ii) $R(K_a)$ is a monotonically non increasing function of K_a , i.e.,

$$1 \geq R(K_{a1}) \geq R(K_{a2}) \geq \dots \geq R(K_{an-1}) \geq 0$$

when

$$0 \leq K_{a1} \leq K_{a2} \leq K_{a3} \dots \leq \infty \quad (A.14)$$

$$(iii) \quad R(0) = 1 \quad \text{and} \quad R(\infty) = 0 \quad (A.15)$$

The Lobatto quadrature formula was chosen because it is exact at the two extreme points and therefore, the last constraint is automatically satisfied. The second constraint drastically narrows down the region of search to that within the confine of two neighbouring points and prevents wild oscillation. In the present study Fibonacci univariate search was used to find out the best values of $L(x_1)$ and therefore $R(K_{a1})$. In cases where $R(K_{a1})$ did not cover the abscissa range of interest, computations were repeated after suitable adjustment of scaling factor s . The optimisation was usually initiated with s equal to unity, and subsequently the scaling factor was changed if found necessary.

In practice, the naturally occurring ores contain very large amounts of non-floatable material. In order to take this into account, the inversion method must be suitably modified. Let M_∞ be the fraction of this non-floatables in the original feed; in this case the Eq. 4.7 becomes

$$M_p(t) = [1 - M_\infty] \int_0^\infty M_p(K_a) \exp(-K_a t) dK_a + M_\infty \quad (A.16)$$

Substitution of Eq. A.1 in the above equation and integration give

$$\bar{M}_p(t) = \int_0^{\infty} \bar{R}(K_a) \exp(-K_a t) dK_a \quad (A.17)$$

where

$$\bar{R}(K_a) = (1 - M_{\infty}) R(K_a) \quad (A.18)$$

Although Eq. A.17 is similar to Eq. A.2, it should be noted that $\bar{R}(0)$ is equal to an unknown $(1 - M_{\infty})$. Thus one of the end points corresponding to $x_n = 1$ in Eq. A.10 is no longer pre-assigned, and therefore, the Lobatto quadrature cannot be used. Eq. A.17 can be transformed into

$$\bar{M}_p(t/s) = \frac{s}{2t} \int_{-1}^1 \bar{R} \left[s \ln \left(\frac{2}{x+1} \right) \right] (x+1)^{t-1} dx \quad (A.19)$$

and the integral can be evaluated using n-point Legendre quadrature formula. This method directly gives the amount of non-floatables in the feed. The abscissas and weights of Legendre quadrature formula are given in Table A.1.

TABLE A.1

Parameters of Lobatto and Legendre Quadrature Formula

Lobatto Quadrature		Legendre Quadrature	
Abscissa	Weight	Abscissa	Weight
1.00000000	0.03571428	0.96028986	0.10122854
0.87174015	0.21070422	0.79666678	0.22238103
0.59170018	0.34112270	0.52553241	0.31370666
0.20929922	0.41245880	0.18343464	0.36268378
-0.20929922	0.41245880	-0.18343464	0.36268378
-0.59170018	0.34112270	-0.52553241	0.31370666
-0.87174015	0.21070422	-0.79666678	0.22238103
-1.00000000	0.03571428	-0.96028986	0.10122854

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